# MMS Project
## Long-Term Integrity of Deepwater Cement Systems Under Stress/Compaction Conditions

## Report 5

Issued September 13, 2003

CSI

CEMENTING SOLUTIONS, INC.

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## Objectives

The overall objective of this project is to determine the properties that affect cement's capability to produce a fluid-tight seal in an annulus. The project primarily focuses on deepwater applications, but general applications will also be examined. The research conducted thus far is focused on the measurement and correlation of cement's mechanical properties to the cement's performance. Also, research was conducted to determine which laboratory methods should be used to establish the cement's key properties.

Results obtained during this reporting period focused on continued measurement of and correlation of cement mechanical properties and mechanical bond integrity of a cemented annulus. Mechanical property testing included measurement of tensile strength and Young's modulus/Poisson's ratio under various confining loads. A new test procedure, anelastic strain/failure testing was begun on several compositions during this project period, and is described in Appendix A. Mechanical integrity testing included shear bond and annular seal testing on specimens cured under various cyclic curing schedules including introduction of intermediate restraint specimens. The results of these tests are tabulated in the Results section below. Additionally, all rock properties test results developed during this project, including graphical data, are presented in Appendix B.

## Observations and Recommendations for Future Work

Results of testing during this reporting period indicate:

- Poisson's ratio data are at least consistent with data from other ongoing testing projects. API is currently examining measurement of Poisson's ration with similar results.
- Measurement of anelastic strain with cycling will allow a more thorough assessment of each composition's performance.
- Intermediate formation strength simulated by PVC pipe is acceptable for mechanical integrity testing to further quantify the performance of the compositions.

Future work includes:

- implementation of test procedure for annular seal and bond strength modifications
- quantification of anelastic strain magnitudes and analysis of consequences in the well environment.
- complete analysis of column sealing tests
- completion of a decision matrix for optimizing cement composition (the final deliverable of this project)

The matrix will be similar in operation to one commissioned by 3M to select optimum lightweight cement for various conditions. This decision matrix will accept well conditions as inputs and will contain performance properties for the various cements tested in the project. A semi-quantitative analysis of the inputs vs. cement performance will allow the user to determine the optimum cement composition for maintaining annular seal under the well conditions.

Plans are to conduct a workshop of steering committee participants in December to complete the decision matrix.

## Testing Program and Procedures

The following cement slurries will be examined: Type 1, foamed cement, bead cement, Class H cement, latex cement. The effect of adding fibers or expansion additives to a slurry will also be examined. The cements are tested primarily for deepwater applications, but their performance under all application conditions is also examined.

Tasks in the project are listed below:

Task 1 – Problem Analysis
Task 2 – Property Determination
Task 3 – Mathematical Analysis
Task 4 – Testing Baseline
Task 5 – Refine Procedures
Task 6 – Composition Matrix
Task 7 – Conduct Tests
Task 8 – Analyze Results
Task 9 – Decision Matrix

Compositions tested in this project are outlined in **Table 1** below. The range of compositions chosen covers the compositions traditionally used in deep water applications as well as newly utilized compositions and compositions designed to produce improved performance.

**Table 1—Cement Compositions for Testing**

| Description | Cement | Additives | Water Requirement (gal/sk) | Density (lb/gal) | Yield ($ft^3$/sk) |
|---|---|---|---|---|---|
| Neat Type I slurry | Type 1 | — | 5.23 | 15.6 | 1.18 |
| Type I slurry with fibers | Type 1 | 3.5% carbon fibers-milled | 5.2 | 15.6 | 1.16 |
| Latex slurry | Type 1 | 1.0 gal/sk LT-D500 | 4.2 | 15.63 | 1.17 |
| Latex slurry with fibers | Type 1 | 1.0 gal/sk LT-D500<br>3.5% carbon fibers-milled<br>0.50% Melkrete | 4.09 | 15.63 | 1.20 |
| Foam slurry (12-lb/gal) | Type 1 | 0.03 gal/sk Witcolate<br>0.01 gal/sk Aromox C-12<br>1% CaCl | 5.2 | 12.0 | 1.19 |
| Bead slurry | Type 1 | 13.19% K-46 beads | 6.69 | 12.0 | 1.81 |
| Neat Class H slurry | Class H | — | 4.3 | 16.4 | 1.08 |
| Class H slurry with fibers | Class H | — | 4.3 | 16.4 | 1.08 |
| Sodium metasilicate slurry | Type 1 | — | 14.22 | 12.0 | 2.40 |

Four major categories of tests are used to analyze the cements: cement design performance testing, mechanical properties testing, mechanical integrity testing, and numerical simulation. Results of mechanical properties testing and mechanical integrity testing are provided in the "Test Results" section of this report, beginning on Page 4.

### *Cement Design Performance*

Standard cement design performance testing, including rheology, thickening time, free fluid, set time, compressive strength, etc. are is performed according to procedures outlined in API Spec. 10.

### *Mechanical Properties*

Mechanical properties tested include: tensile strength/tensile Young's modulus (T), compressive Young's modulus, Poisson's ratio, and anelastic strain-fatigue testing.

The tensile strengths are determined with the Brazilian Test Method. From this test, the tensile Young's modulus is computed, as well as the maximum yield of the slurry.

The compressive Young's modulus will be determined through compression tests with confining loads (defined by 0 psi break) with a baseline of a 14-day cure. Poisson's ratio will also be determined from these tests, Poisson's ratio values will vary with respect to the stress rate, slurry type, air entrainment, and perhaps other variables.

Anelastic strain and fatigue testing is a modification of hydrostatic testing. The modified procedure involves cycling samples repeatedly to 25%, 50%, and 75% of each composition's compressive strength under 500-psi confining stress. Measurement of anelastic strain with cycling should provide a more comparable measure of each composition's performance.

### *Mechanical Integrity*

The mechanical integrity issues of the cement slurries include stresses in the cement, and the flow of fluids around the cement and through the matrix of the cement. To predict the flow of fluid around the cement, the cement slurries will be tested for bonding capacity, presence of microannuli, and deformation. The flow of fluids through the matrix of the cement will be examined through tests for detecting cracks and permeability changes. The stress undertaken by the cement slurries will be determined as a function of pressure, temperature, pipe buckling, and formation compaction. Stresses under cyclic conditions will also be determined.

Shear bond and annular seal measurements are taken under cyclical conditions for both soft and hard formations. Results from testing with simulations of hard and soft formations indicate the need for a simulated formation of intermediate strength. The altered shear and annular seal testing will include a simulated medium-strength formation with Schedule 40 PVC pipe as the outside mold for the cement sheath.

Additional stresses will be imposed on all test specimens by increasing the maximum pressure to which the inner pipe is stressed. Additionally, shear bond tests will be run only after a composition has been tested for annular seal. The shear bond test samples will be subjected to the same pressure and temperature cycling that produced annular seal failure before shear bond is evaluated. This procedure will provide a comparison between shear bond and annular seal behavior.

Cement column seal tests illustrate the sealing effectiveness of several cements that are subjects of the project. These tests are designed to test a cement's capacity to isolate gas pressure across an enclosed column. Ten-foot lengths of 2-in. pipe are filled with cement slurry, pressurized to 1000 psi, and then cured for 8 days. After the test samples have cured, low-pressure gas (100 to 200 psi) is periodically applied to one end of each test pipe and the gas flow rate through the cement column is measured. As time increases with no flow, increased pressure is applied to the pipe to eventually induce failure and flow.

## Test Results—Mechanical Properties

This section contains results from testing conducted throughout this project period, as well as additional mechanical property test results selected from previous test periods. Graphical data for all mechanical property tests are presented in Appendix B of this report.

## Tensile Strength

Table 2 shows the effects of carbon fibers on tensile strength. The two-fold to three-fold increase in tensile strength is significant, indicating the potential for fibers to enhance the durability of cement.

**Table 2—Tensile Strength and Young's Modulus**

| Slurry | Tensile Strength (psi) | Young's Modulus |
|---|---|---|
| Foam slurry (12-lb/gal) | 253 | 3.23 E4 |
| Neat Type I slurry | 394/213[a] | 19.15/8.16 E4[a] |
| Type I slurry with fibers | 1071 | 9.6 E4 |
| Latex slurry | 539 | 5.32 E4 |
| Latex slurry with fibers | 902 | 8.5 E4 |

[a]Data taken from two different specimens.

## Young's Modulus with Various Confining Forces

The effects of confining stress on compressive strength and Young's modulus are presented in Table 3. A significant increase in compressive strength is observed among lower-strength compositions such as foam cement and latex cement, as confining stress is increased.

**Table 3—Young's Modulus at Various Confining Stresses**

| Slurry Composition | Confining Pressure (psi) | Young's Modulus (psi) |
|---|---|---|
| Type I slurry | 0 | 16.7 E 5 |
| | 1500 | 11.1 E 5 |
| | 5000 | 9.1 E 5 |
| Foam slurry (12 lb/gal) | 0 | 5.8 E 5 |
| | 500 | 6.8 E 5 |
| | 1000 | 6.1 E 5 |
| Bead slurry (12 lb/gal) | 0 | 9.5 E 5 |
| | 500 | 8.1 E 5 |
| | 1000 | 1 E 6 |
| Latex slurry | 0 | 5.6 E 5 |
| | 250 | 8.9 E 5 |
| | 500 | 9.4 E 5 |

## Poisson's Ratio

Initial results of Poisson's ratio testing on these lightweight cement compositions were unexpectedly low. Continued Poisson's ratio testing during this test period to determine reasons for these low values confirmed the accuracy of these early results. The low Poisson's ratio values for these compositions are theorized to be related to the porosity of the specimens. Several published technical reports have documented this tendency for Poisson's ratio to be effectively lowered as porosity increases.

Another potential variable in Poisson's ratio testing is load rate. An investigation into the effect of load rate on Poisson's ratio indicated that load rate does affect Poisson's ratio measurement (Table 4). Table 5 presents data generated with a load rate of 250 psi/min. While these values are lower than what has traditionally been considered acceptable, the data are generally positive.

CT scans performed on Poisson's ratio test specimens indicated a link between large voids or pore spaces and variable Poisson's ratio. This procedure will be included in future testing and samples with large voids will be discarded. CT scans are included in Appendix B.

**Table 4—Effect of Load Rate on Poisson's Ratio**

| Load Rate | Poisson's Ratio |
|---|---|
| 100 psi/min | 0.1 |
| 250 psi/min | 0.08 |
| 500 psi/min | -0.01 |

**Table 5—Poisson's Ratio**
**(50-psi confining pressure, 250 psi/min load rate)**

| Slurry | Failure (psi) | Poisson's Ratio |
|---|---|---|
| Foam slurry (12-lb/gal) | 3100 | 0.00 |
| Bead slurry | 4100 | -0.01 |
| Neat Class H slurry | 6450 | 0.0012 |
| SMS slurry | 920 | 0.005 |
| Type I slurry | 6500 | 0.1 |

### *Anelastic Strain*

Anelastic strain testing is a variation of hydrostatic testing and is designed to allow a more accurate evaluation of permanent strain resulting from stressing different test compositions. This procedure standardizes confining stress at 500 psi and calls for samples to be cycled to 25%, 50%, and 75% of each composition's compressive strength under that confining stress. Measurement of anelastic strain with cycling provides a more comparable value of each composition's performance.

Results of initial anelastic strain testing are presented in Table 6. Strain data are reported as final strain minus initial strain measurements, with final being at the end of three cycles. A point on the stress axis indicating minimum linear strain was picked for comparison of strains at the beginning and end of cycling. This comparison point is listed also. Data generation will continue and will include a round of samples tested to a common stress maximum to provide two alternate methods of comparison.

**Table 6—Results of Anelastic Strain Testing**

| Composition | Failure (psi) | Comparison Stress (psi) | Strain (mm/mm) | |
|---|---|---|---|---|
| | | | 25% | 50% |
| Type I slurry | 6000 | 600 | 0.0006 | 0.0007 |
| Foam slurry | 2000 | 300 | 0.001 | — |
| Bead slurry | 3300 | 400 | 0.0007 | — |
| Class H slurry | 6000 | 600 | 0.0007 | 0.0009 |

# Test Results—Mechanical Integrity

This section contains results from testing conducted throughout this project period, as well as additional mechanical integrity test results selected from previous test periods.

### *Shear Bond*

Results of shear bond testing (Table 7) indicated that bond was degraded extensively both by pressure and temperature cycling. This degradation seemed to be aggravated by the simulated soft formation. In future tests, a modified shear bond method will be used to help ensure that the results are more comparable to annular seal tests. Shear bond testing simulating intermediate formation strength with PVC pipe was initiated, and a successful beta test has been completed. It is anticipated that intermediate formation strength will be completed during the next test period.

**Table 7—Shear Bond Strengths (psi)**

| System | Simulated Formation | Type I Slurry | Foam Slurry | Bead Slurry | Latex Slurry |
|---|---|---|---|---|---|
| Baseline | hard | 1194 | 127/98 | 109/78 | — |
| | soft | 198 | 233 | 143 | 223 |
| Temperature-Cycled | hard | 165 | 299/215 | 191/269 | — |
| | soft | 72 | 7 | 56 | 149 |
| Pressure-Cycled | hard | 194/106 | 276/228 | 294/170 | — |
| | soft | 23 | 22* | 23* | 11 |

* Visual inspection revealed samples were cracked.

### *Annular Seal*

Results presented in Table 8 indicate that all cyclic testing specimens failed in the soft formation simulation while all specimens in the hard-formation tests maintained seal. These results indicate the need for a simulated formation with intermediate strength to further differentiate seal effectiveness. Additional stresses for the hard-formation simulation must be imposed through application of heat or pressure. .

A series of annular seal tests was performed with the intermediate strength formation simulated by PVC pipe. Results with Type 1 cement indicated failure after the third temperature cycle. Unfortunately, problems with flow meter calibration caused the quantitative data to be worthless. This testing will be repeated for all cement compositions.

**Table 8—Annular Seal Tests**

| Condition Tested | Formation Simulated | Type I Slurry | Foamed Slurry | Bead Slurry |
|---|---|---|---|---|
| Initial Flow | Hard | 0 Flow | 0 Flow | 0 Flow |
| | Soft | 0 Flow | 0.5 (md) | 0 Flow |
| Temperature-Cycled | Hard | 0 Flow | 0 Flow | 0 Flow |
| | Soft | 0 Flow | 123 md | 43 md* |
| Pressure-Cycled | Hard | 0 Flow | 0 Flow | 0 Flow |
| | Soft | 27 md | 0.19 md* | 3 md |

* Visual inspection revealed samples were cracked.

### *Cement Column Seal*

Four duplicate sets of models were filled with cement compositions listed in Table 9.

**Table 9—Compositions Tested for 8-ft Permeability Models**

| Composition | Density (lb/gal) | Yield ($ft^3/sk$) | Water (gal/sk) | Columns |
|---|---|---|---|---|
| Type I slurry | 15.6 | 1.18 | 5.23 | 1 and 2 |
| SMS slurry | 12 | 2.38 | 14.05 | 3 and 4 |
| Bead slurry | 12 | 1.81 | 6.69 | 5 and 6 |
| Latex slurry | 15.63 | 1.17 | 4.20 | 7 and 8 |

These cements were allowed to cure for 7 days, and were then tested with differential pressure as described in the procedure section. Results, summarized in Table 10, are for days tested after the initial curing period. Actual results are shown in Appendix B, Table B1, page 34.

**Table 10—Failure of 8-ft Permeability Models**

| Column | Days Tested at Initial Flow | Differential (psi) | Flow Rate (cc/min) |
|---|---|---|---|
| 1 | 107 | 500 | 0.09 |
| 2 | 51 | 200 | 0.1 |
| 3 | 1 | 100 | 33 |
| 4 | 1 | 100 | 26 |
| 5 | 78 | 400 | 0.03 |
| 6 | 84 | 400 | 0.02 |
| 7 | 84 | 400 | 0.02 |
| 8 | 99 | 500 | 3.1 |

These results indicate that the sodium metasilicate (SMS) cement failed very quickly on the first day of testing. Other compositions including the neat Type 1 cement required up to 500 psi over the 8-ft column to induce failure. Further analysis of the complete data will be performed to determine bulk permeability with time.

# Appendix A—Test Procedures

### Sample Preparation

Some preparation and testing methods were modified to adapt for the lightweight bead and foamed slurries. The mixing procedures for the bead slurry were also modified to minimize bead breakage due to high shear from API blending procedures. The following blending procedure was used for the bead slurry.

1. Weigh out the appropriate amounts of the cement, water, and beads into separate containers.
2. Mix the cement slurry (without beads) according to Section 5.3.5 of API RP 10B.
3. Pour the slurry into a metal mixing bowl and slowly add beads while continuously mixing by hand with a spatula. Mix thoroughly.
4. Pour this slurry back into the Waring blender and mix at 4,000 rev/min for 35 seconds to mix and evenly distribute the contents.

Testing methods for the foamed slurries were also modified. For example, thickening time is performed on unfoamed slurries only. Because the air in the foam does not affect the hydration rate, the slurry is prepared as usual per API RP 10B and then the foaming surfactants are mixed into the slurry by hand without foaming the slurry.

### Sample Curing

Test specimens for rock properties testing are mixed in a Waring blender and poured into cylinder molds. Samples are cured for 7 days in a 45°F atmospheric water bath.

Performance test fixture molds are filled with cement mixed in the same manner. These fixtures are also cured in a 45°F water bath for 7 days prior to testing.

### Thickening Time Test

Following the procedures set forth in API RP 10B[1], thickening-time tests were performed on the three cement systems. The test conditions started at 80°F and 600 psi, and were ramped to 65°F and 5,300 psi in 48 minutes.

### Free-Fluid Test

The free-fluid testing that was performed on the Type 1, foamed cement and bead cement came from API RP 10B. The free-fluid procedure, also referred to as operating free water procedure, uses a graduated cylinder that is oriented vertically. The slurry is maintained at 65°F, and the free fluid that accumulates at the top of the slurry is measured. See Table A1 for test results.

**Table A1—Free Fluid Test Results**

| Slurry System | Thickening Time to 100 Bc (hr:min) | Percentage of Free Fluid |
|---|---|---|
| Neat | 4:38 | 0.8 |
| Foamed | 3:42 | 0.0 |
| Bead | 5:04 | 0.8 |

### *Compressive Strength*

The compressive strengths were derived using the 2-in. cube crush method specified in API RP 10B. The samples were cured in an atmospheric water bath at 45°F. The reported values were taken from the average of three samples.

### *Young's Modulus and Poisson's Ratio Testing*

Traditional Young's modulus testing was performed using ASTM C469[2], Standard Test Method for Static Modulus of Elasticity (Young's Modulus) and Poisson's Ratio of Concrete in Compression.

The following procedure is used for the Young's modulus testing.

1. Each sample is inspected for cracks and defects.
2. The sample is cut to a length of 3.0 in.
3. The sample's end surfaces are then ground to get a flat, polished surface with perpendicular ends.
4. The sample's physical dimensions (length, diameter, weight) are measured.
5. The sample is placed in a Viton jacket.
6. The sample is mounted in the Young's modulus testing apparatus.
7. The sample is brought to 100-psi confining pressure and axial pressure. The sample is allowed to stand for 15 to 30 min until stress and strain are at equilibrium. (In case of an unconfined test, only axial load is applied.)
8. The axial and confining stress are then increased at a rate of 25 to 50 psi/min to bring the sample to the desired confining stress condition. The sample is allowed to stand until stress and strain reach equilibrium.
9. The sample is subjected to a constant strain rate of 2.5 mm/hr.
10. During the test, the pore-lines on the end-cups of the piston are open to atmosphere to prevent pore-pressure buildup.

After the sample fails, the system is brought back to the atmospheric stress condition. The sample is removed from the cell and stored.

Following a review of this procedure during the February meeting, the decision was made to conduct additional load tests in the constant stress mode rather than the constant strain mode.

### *Hydrostatic Cycling and Anelastic Strain*

Hydrostatic cycling testing was then performed on cement specimens in the same load configuration as for Young's modulus and Poisson's ratio. This testing was conducted with axial loading and radial loading being maintained equally throughout the load ramping process. For such testing, the hydrostatic pressure is cycled through the following ramping procedures.

1. Ramp up to 1,000 psi.
2. Ramp down to 100 psi.
3. Ramp up to 1,500 psi.
4. Ramp down to 100 psi.
5. Ramp up to 2,000 psi.
6. Ramp down to 100 psi.
7. Continue to failure.

Each ramp was conducted at 16.7 psi/min and the sample was held at the destination hydrostatic pressures (i.e., 100; 1,000; 1,500; and 2,000 psi) for no longer than two minutes before proceeding to the next ramp step.

Hydrostatic cycling was studied further to investigate the deformation that occurs during each of the ramps. The value (size) of the sample at 250 psi during the first ramp to 1,000 psi is the reference value for determining the percentile of deformation. This reference value (sample size) is then compared to the sample size at 250 psi during each subsequent ramp step.

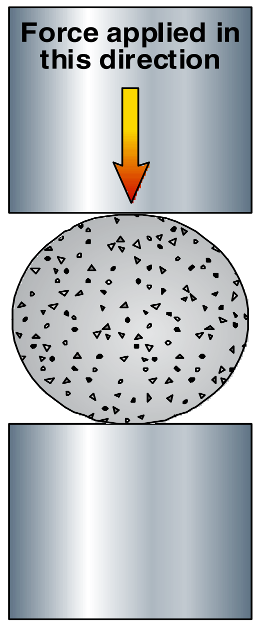
Concern over the ability to compare results of this testing among different compositions led to the development of a test for determining strain and cyclic loading effects under similar conditions with respect to each composition's ultimate strength. This test is referred to as anelastic strain testing.

Anelastic strain testing, a variation of hydrostatic testing, is designed to allow a more accurate evaluation of permanent strain resulting from stressing different test compositions. Samples are cycled to 25%, 50%, and 75% of each composition's compressive strength under 500-psi confining stress. Measurement of anelastic strain with cycling provides a more comparable value of each composition's performance. The first step in the procedure involves compression testing a sample to failure in the load cell with 500-psi confining stress. Once this failure load value is determined, additional samples will be tested by applying axial loads equal to 25%, 50%, and 75% of the failure load, and cycling until samples fail. The cyclic loading rate will be maintained at 250 psi/min and the confining force will be maintained at 500 psi. Plastic deformation will be measured at the end of each cycle. Results will include cycles to failure and anelastic strain per cycle. CT scans will be performed on each sample prior to testing to rule out the presence of any large voids.

### *Tensile Strength and Tensile Young's Modulus*

Tensile strength was tested using ASTM C496[3] (Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens). For this testing, the specimen dimensions were 1.5 in. diameter by 1 in. long. **Figure A1** shows a general schematic of how each specimen is oriented on its side during testing. The force was applied by constant displacement of the bottom plate at a rate of 1 mm every 10 minutes. Change in the specimen diameter can be calculated from the test plate displacement. The (compressive) strength of the specimen during the test can be graphed along with the diametric strain (change in diameter/original diameter) to generate the tensile Young's modulus.

**Figure A1—Sample Orientation for ASTM C496-90 Testing**




### *Annular Seal Testing Procedure*

Samples for annular seal testing are prepared by mixing cement compositions, pouring them into specified molds, and curing them for 7 days in 45°F water baths. After curing, three specimens for each test composition and condition are tested.

These procedures are for use with the annular seal apparatus. Specific procedures are applied as necessary for each formation simulation: soft, intermediate, and hard. The soft apparatus test procedure is to be used with cores cured to set in a soft gel mold, which provides a semi-restricting force on the outside of the core. The intermediate specimen mold uses a 3-in. diameter Schedule 40 PVC pipe as the outer containment. The hard apparatus uses a 3-in. Schedule 40 steel pipe as the outside containment, giving the cement slurry a restricting force outside of the core.

**Soft-Formation Simulation**

1. After the core is cured, place the core inside the gel mold sleeve.
2. Place the core and sleeve inside the pipe-in-soft steel cell.
3. Once inside, both ends of the core are supported with O-rings.

4. The O-rings are then tightened by interior end plates to close off leaks that might be present.
5. Using water, pressurize the exterior circumference of the sleeve to 25 psi and check for leaks on the ends of the cell.
6. Cap off both ends of the steel cell with the cell end caps. One end cap has a fitting that allows for $N_2$ gas to be applied into the cell, and the other end cap allows gas to exit the cell.
7. Attach the pressure inlet line to the bottom of the cell and attach the pressure outlet line to the top of the cell.
8. Apply pressure to the inlet line (do not exceed 20 psig) and measure the flow out using flow meters.

**Hard-Formation Simulation**

1. After the core is cured inside the steel pipe, cap off each end of the pipe with steel end caps. Each end cap has a fitting that allows for gas to be applied into the pipe or to exit the pipe.
2. Attach the pressure inlet line to the bottom of the pipe, and attach the pressure outlet line to the top of the pipe.
3. Apply pressure to the inlet line (do not exceed 20 psig) and measure the pressure out of the outlet line using flow meters.

**Intermediate Formation Simulation**

The test fixture for performing tests with a simulated intermediate formation is very similar to that used for tests with simulated hard formations, except the outer pipe is made of Schedule 40 PVC. Stress is applied to the specimens by applying hydraulic pressure or heat to the inner pipe.

Thermal cycling resulted from the insertion of heaters into the inner pipe and the heating of the inner pipe from 80° to 180°F then allowing the pipe to cool to 80°F. Flow through the model was measured at each endpoint on the cycle, and cycles were repeated a minimum of five times per sample. Three specimens of each composition were tested.

To ensure that sufficient stress could be applied to induce failure in all samples, the thermal cycling test procedure was modified to allow use of a thicker-walled inner pipe that provides more steel volume for expansion. The modified test fixture now features an inside pipe with a 1.68-in. outside diameter and a 1.25-in. inside diameter, giving a wall thickness of 0.190 in. Additionally, the outer containment diameter will be increased to 3 in.

Pressure cycling resulted from the application of hydraulic pressure to the inner pipe. For the initial cycle, pressure was increased from 0 to 500 psi. Pressure was then released and allowed to return to 0, and flow measurements were made. Additional cycles were made by increasing the upper pressure limit by 500 psi (0 to 1,000 to 0 psi, 0 to 1,500 to 0 psi, etc.) and measuring flow at the endpoint (0) of each cycle. If specimens were cycled to 10,000 psi without failure, the 0 to 10,000 to 0 psi pressure cycle was repeated a minimum of five times. The original test procedure was modified to establish a maximum pressure of 10,000 psi during pressure cycles.

### *Shear Bond Strength Testing*

Shear bond strength tests are used for investigating the effect that restraining force has on shear bond. Samples are cured in a hard-formation configuration (**Figure A2**) and in a soft-formation configuration (**Figure A3**). The hard-formation configuration consists of a sandblasted internal pipe with an outer diameter (OD) of 1 $^{1}/_{16}$ in. and a sandblasted external pipe with an internal diameter (ID) of 3 in. Both pipes are 6 in. long. A contoured base and top are used to center the internal pipe within the external pipe. The base extends into the annulus 1 in. and cement fills the annulus to a height of 4 in. The top inch of annulus contains water.

For the soft-formation shear bond tests, plastisol is used to allow the cement to cure in a less-rigid, lower-restraint environment. Plastisol is a mixture of a resin and a plasticizer that creates a soft, flexible substance. This particular plastisol blend (PolyOne's Denflex PX-10510-A) creates a substance with a hardness of 40 duro.

The soft formation configuration contains a sandblasted external pipe with an ID of 4 in. A molded plastisol sleeve with an ID of 3.0 in. and uniform thickness of 0.5 in. fits inside the external pipe. With the aid of a contoured base and top, a sandblasted internal pipe with an OD of 1 $^{1}/_{16}$ in. is then centered within the plastisol sleeve. The pipes and sleeve are 6 in. long. The base extends into the annulus 1 in. and cement fills the annulus to a height of 4 in. between the plastisol sleeve and the inner 1 $^{1}/_{16}$ -in. pipe. The top inch of annulus is filled with water.

The intermediate formation test fixture will feature the same configuration as the hard formation fixture except the outer pipe is made of PVC.

Cycling tests for the shear bond specimens were performed according to the following test schedules:

**Pressure Cycling**

1. Cure specimens for 14 days at 45°F.
2. Apply 5000 psi hydraulic pressure to inner pipe and maintain for 10 minutes.
3. Release and maintain for 10 minutes.
4. Repeat the cycle four more times.
5. Test shear bond.

**Temperature Cycling**

1. Cure specimens for 14 days at 45°F.
2. Move specimens from 45°F water bath to 96°F for 1 hour.
3. Place specimens in 180°F water bath for 4 hours.
4. Place specimens in 96°F water bath for 1 hour.

5. Return specimens to 45°F bath
6. Repeat the cycle four more times.
7. Test shear bond.

If additional shear bond testing is required, a new test procedure will be used that is designed to allow correlation with annular seal test results. After failure is noted in the annular seal test, the exact pressure or temperature cycle sequence is repeated for the shear bond specimens. Shear bond will be measured after the cycling to determine the level of bond remaining.

**Figure A2—Cross-section of pipe-in-pipe test fixture configuration for shear bond test.**

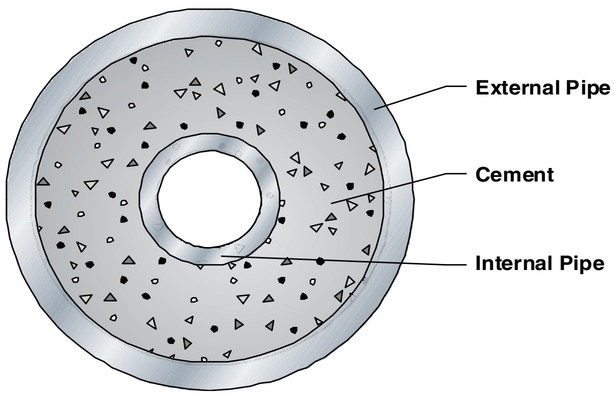
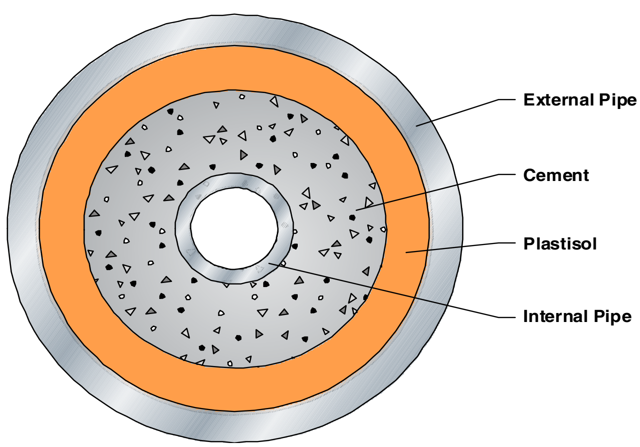
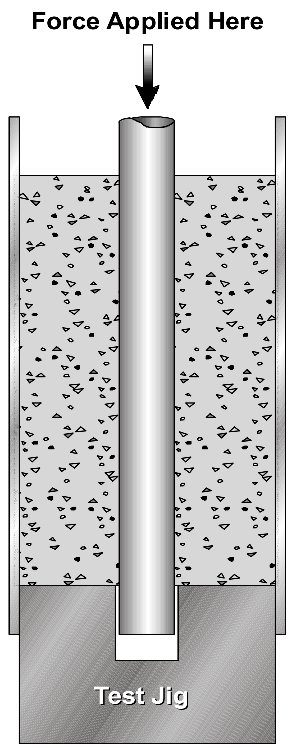



**Figure A3—Cross-section of pipe-in-soft test fixture configuration for shear bond test.**



The shear bond measures the stress necessary to break the bond between the cement and the internal pipe. This was measured with the aid of a test jig that provides a platform for the base of the cement to rest against as force is applied to the internal pipe to press it through. (Figure A4) The shear bond force is the force required to move the internal pipe. The pipe is pressed only to the point that the bond is broken; the pipe is not pushed out of the cement. The shear bond strength is the force required to break the bond (move the pipe) divided by the surface area between the internal pipe and the cement.

**Figure A4—Test jig for testing shear bond strength**




### *Cement Column Seal Tests*

Eight-foot lengths of 2-in. Schedule 40 pipe are mounted vertically and fitted at the top and bottom with end caps equipped with pressure inlet and outlet ports. The bottom of each pipe is filled with 6 in. of 20-40 sand to provide an open base for gas injection. Sets of two fixtures are each filled with one of four different cement slurries: bead, Type 1, latex, and sodium metasilicate. Samples are covered with water and cured for 7 days under 1000-psi pressure. After the samples are cured, 100 psi of pressure is applied to the bottom of each fixture and any flow through the column is monitored.

# Appendix B—Test Data

Graphical data for all mechanical properties tests performed in this investigation are presented in this appendix.

**Figure B1—Plot of tensile strength and Young's modulus results for latex slurry with fibers (sample 1), Type 1 slurry with fibers (sample 2), and latex slurry (sample 3.**

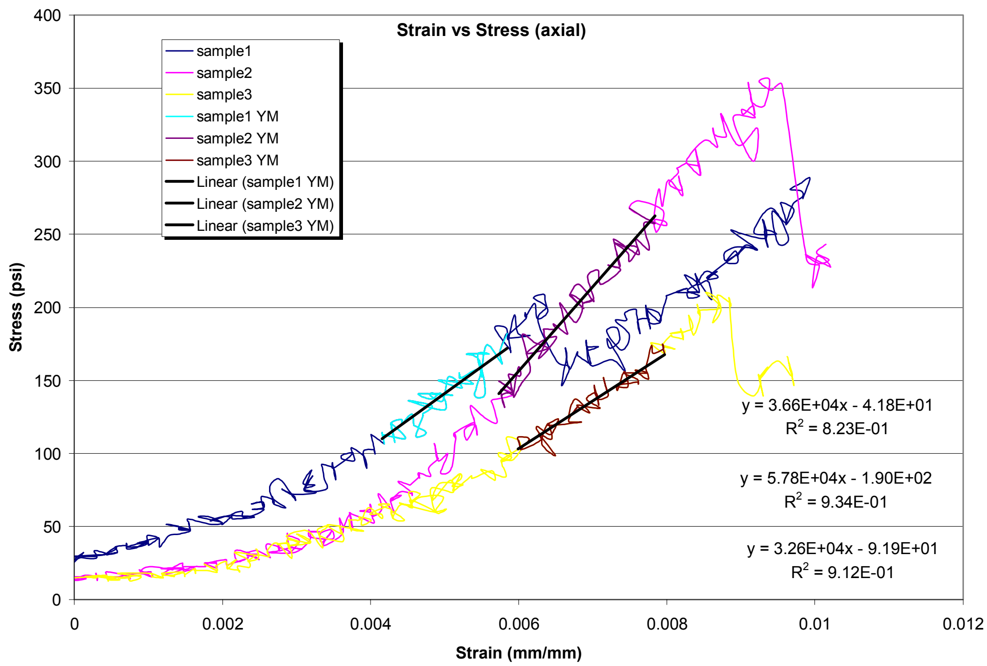


**Figure B2—Plot of tensile strength and Young's modulus results for neat Type 1 slurry cured in a confined state.**

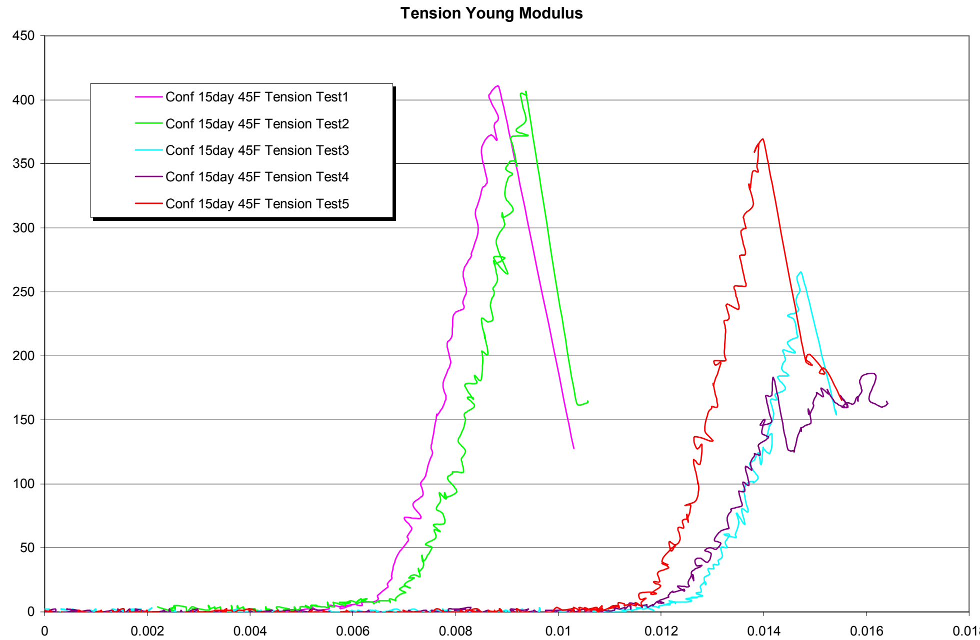



**Figure B3—Plot of tensile strength and Young's Modulus results for 12-lb/gal foam slurry.**

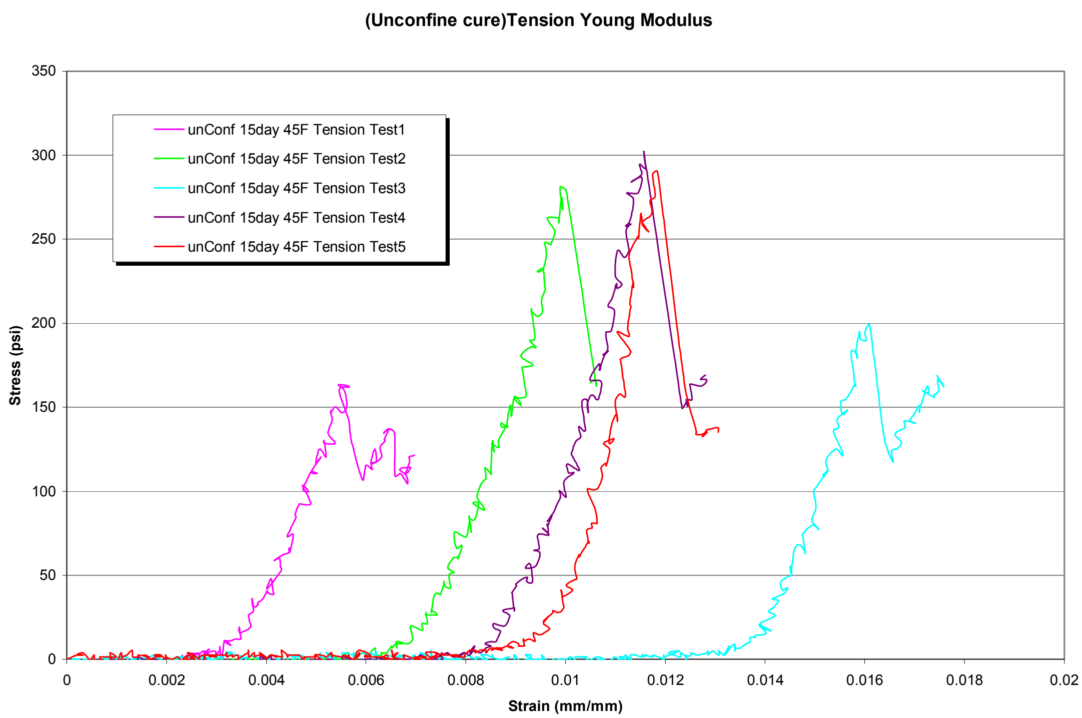


**Figure B4—Plot of compressive Young's modulus for Type 1 slurry at 0-psi confining pressure.**

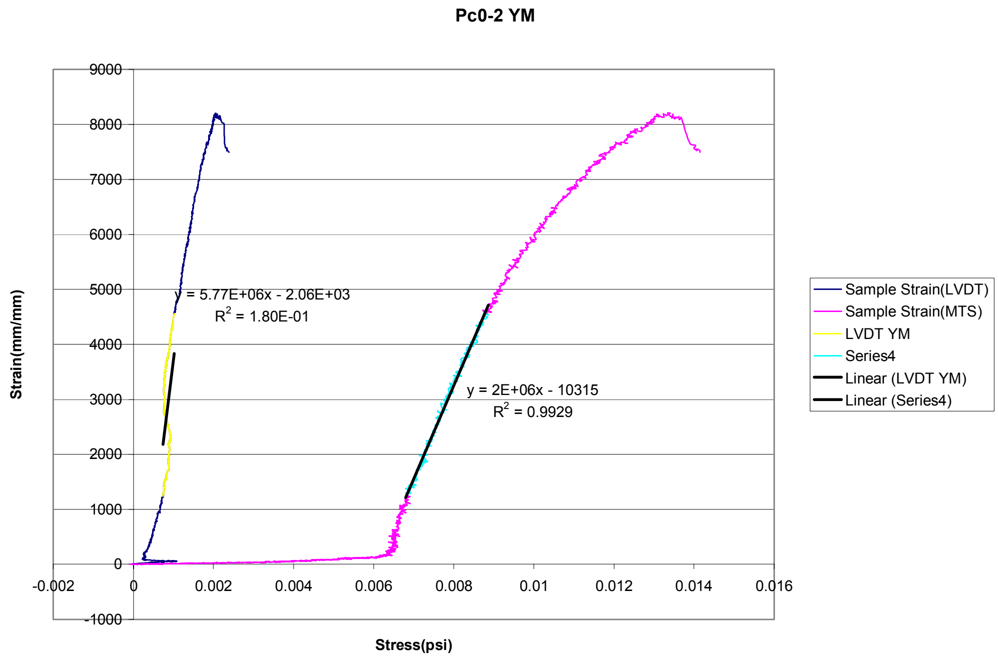



**Figure B5—Plot of compressive Young's modulus for Type 1 slurry at 1500-psi confining pressure.**

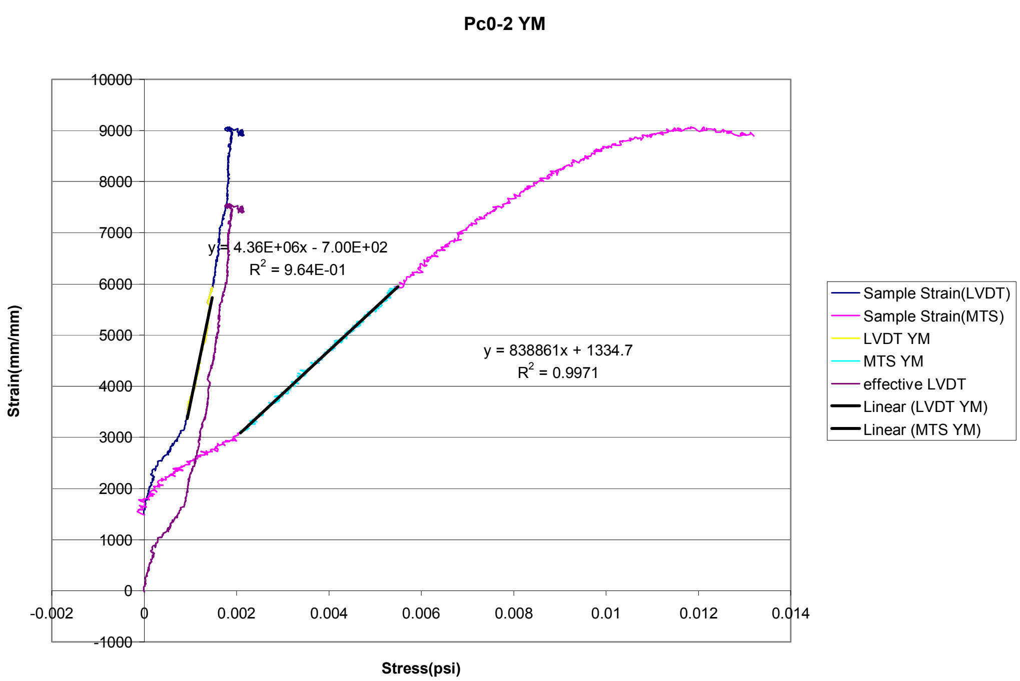


**Figure B6— Plot of compressive Young's modulus for Type 1 slurry at 5000-psi confining pressure.**

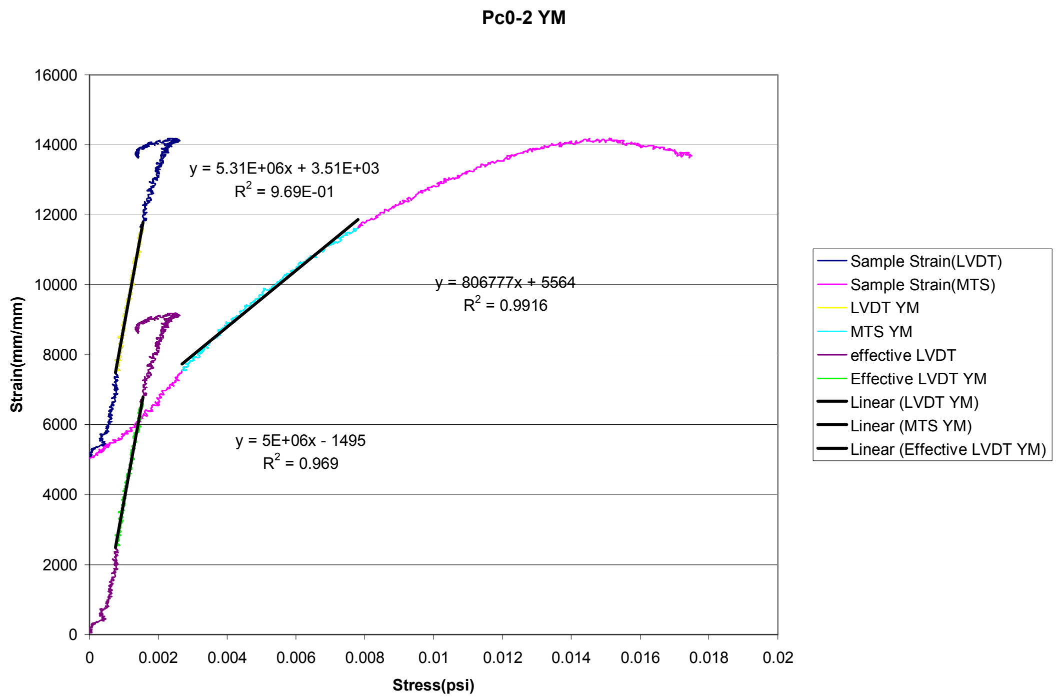



**Figure B7— Plot of compressive Young's modulus for 12-lb/gal foam slurry at 0-psi confining pressure.**

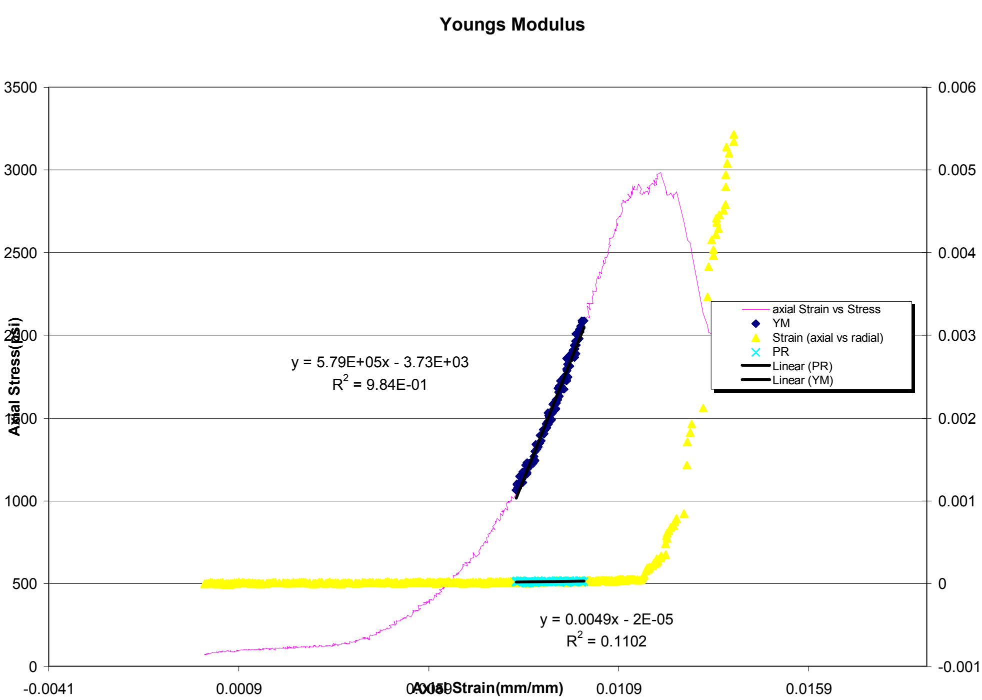


**Figure B8— Plot of compressive Young's modulus for 12-lb/gal foam slurry at 500-psi confining pressure.**

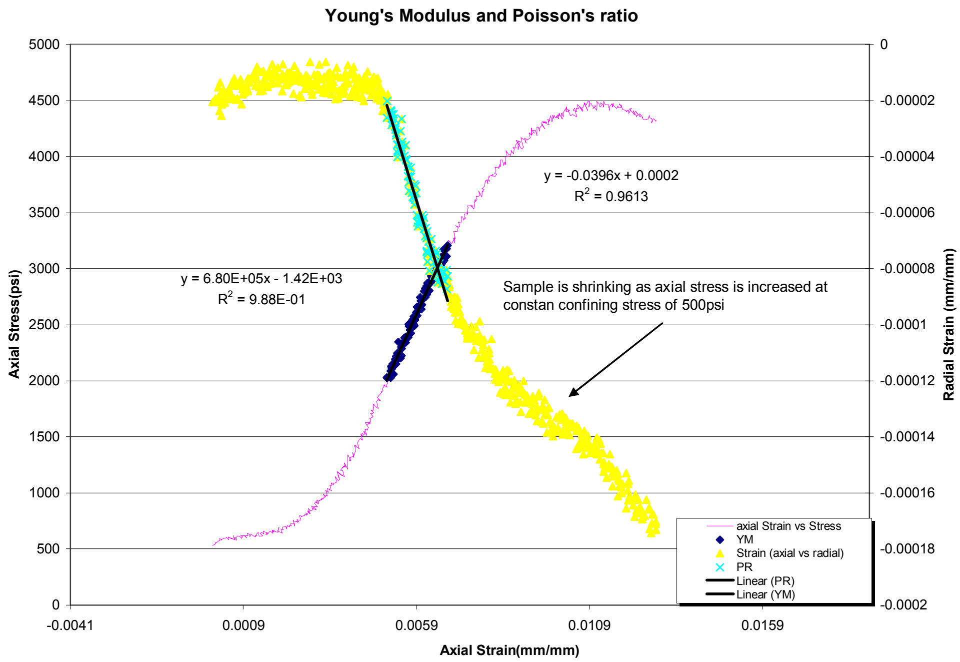



**Figure B9— Plot of compressive Young's modulus for 12-lb/gal foam slurry at 1000-psi confining pressure.**

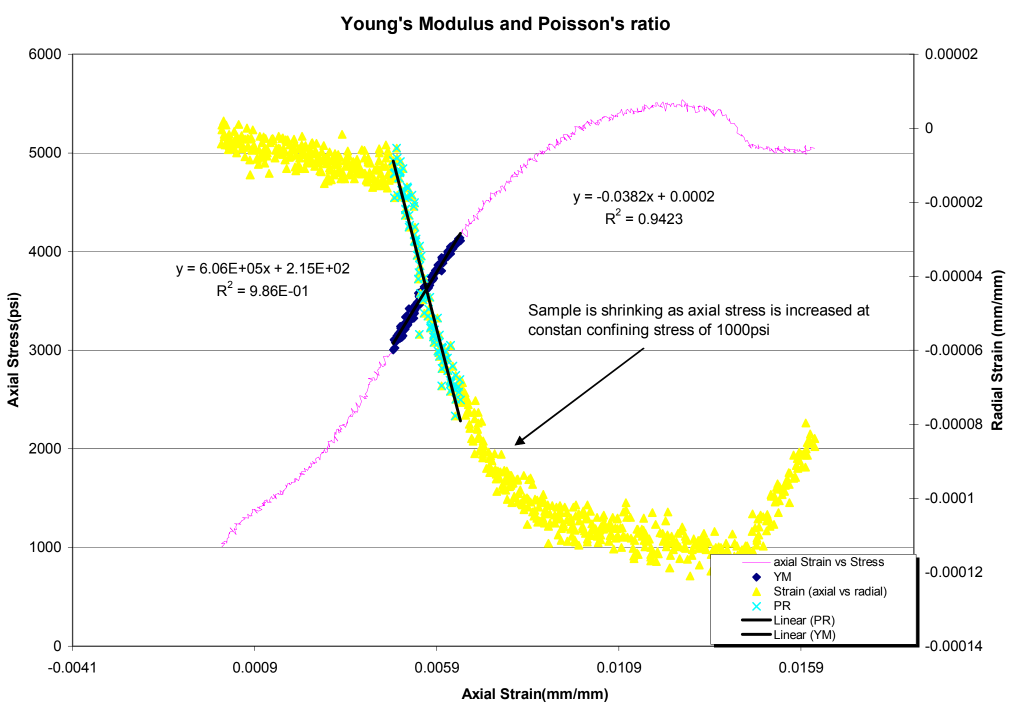


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**Figure B10— Plot of compressive Young's modulus for bead slurry at 0-psi confining pressure.**

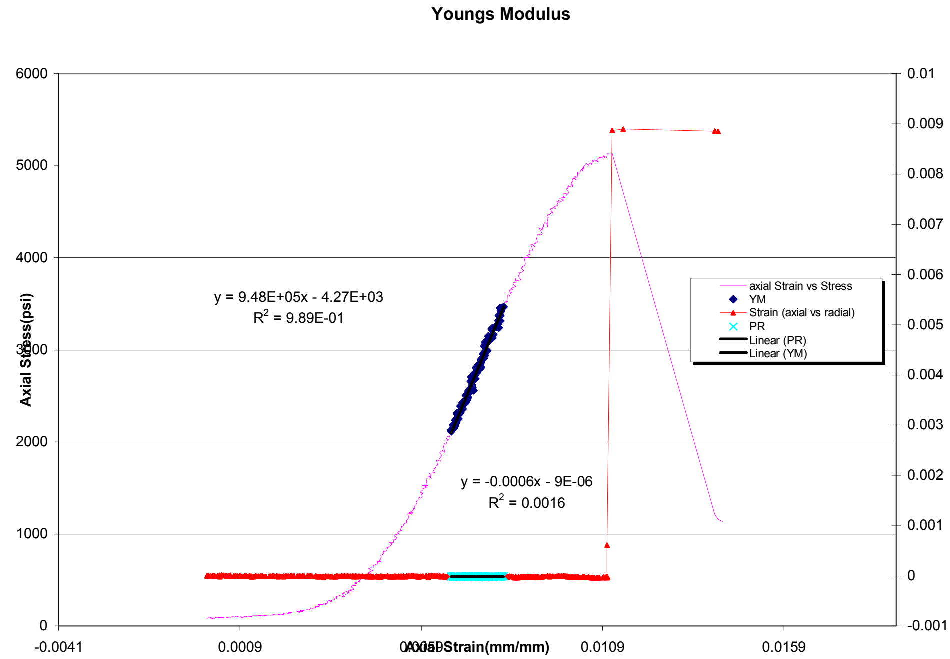



**Figure B11— Plot of compressive Young's modulus for bead slurry at 500-psi confining pressure.**

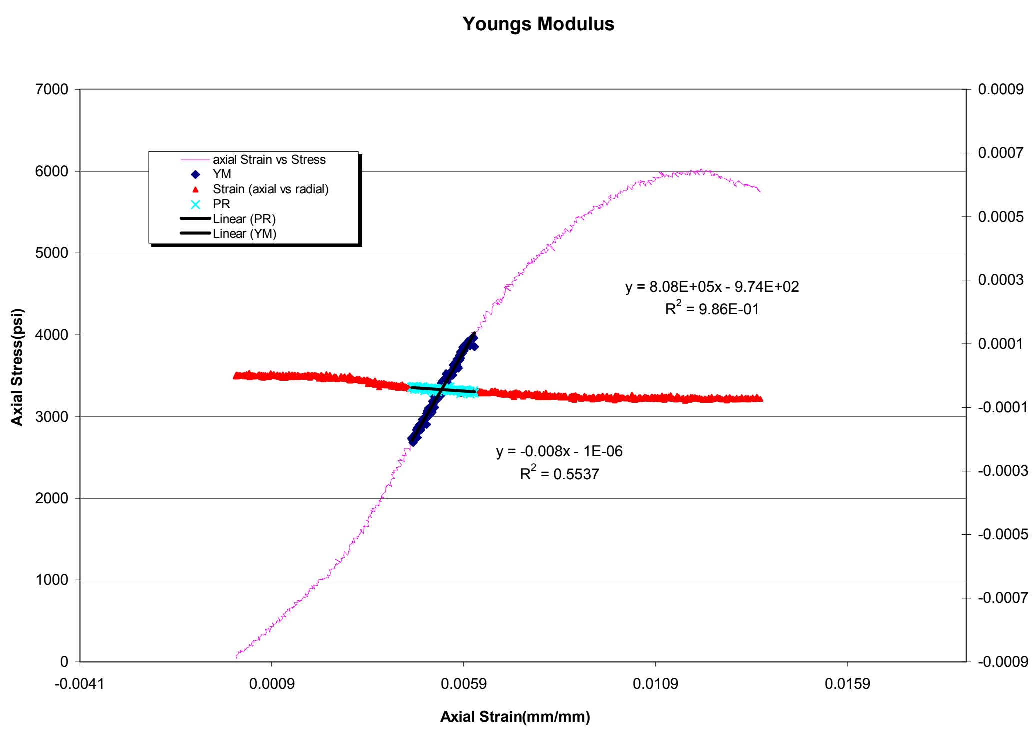


**Figure B12— Plot of compressive Young's modulus for bead slurry at 1000-psi confining pressure.**

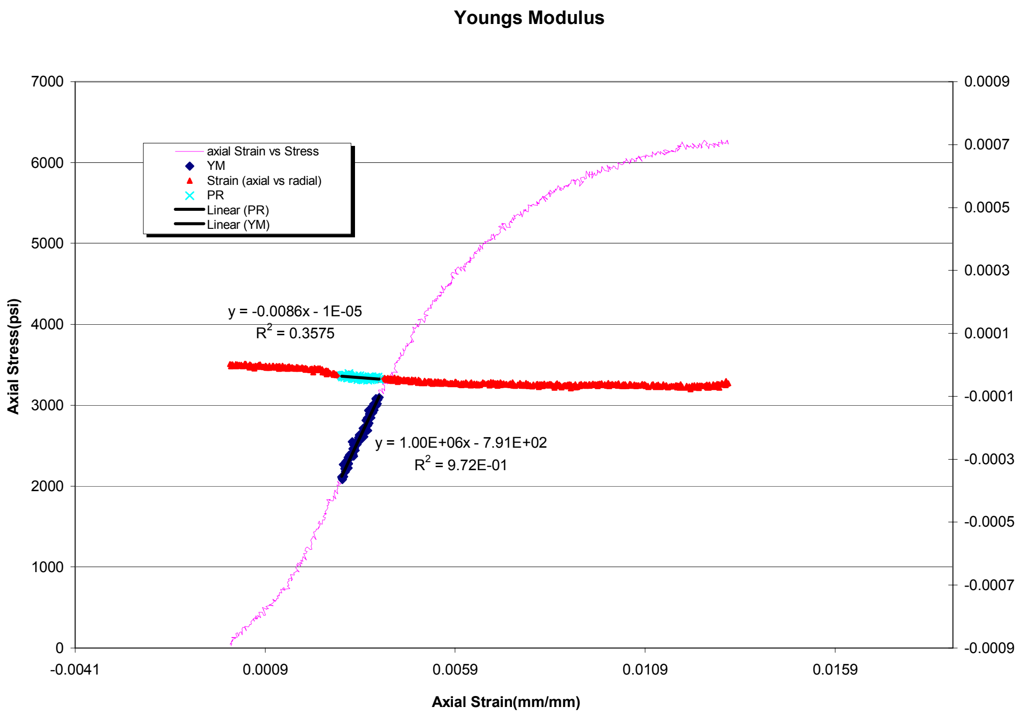



**Figure B13— Plot of compressive Young's modulus for latex slurry at 0-psi confining pressure.**

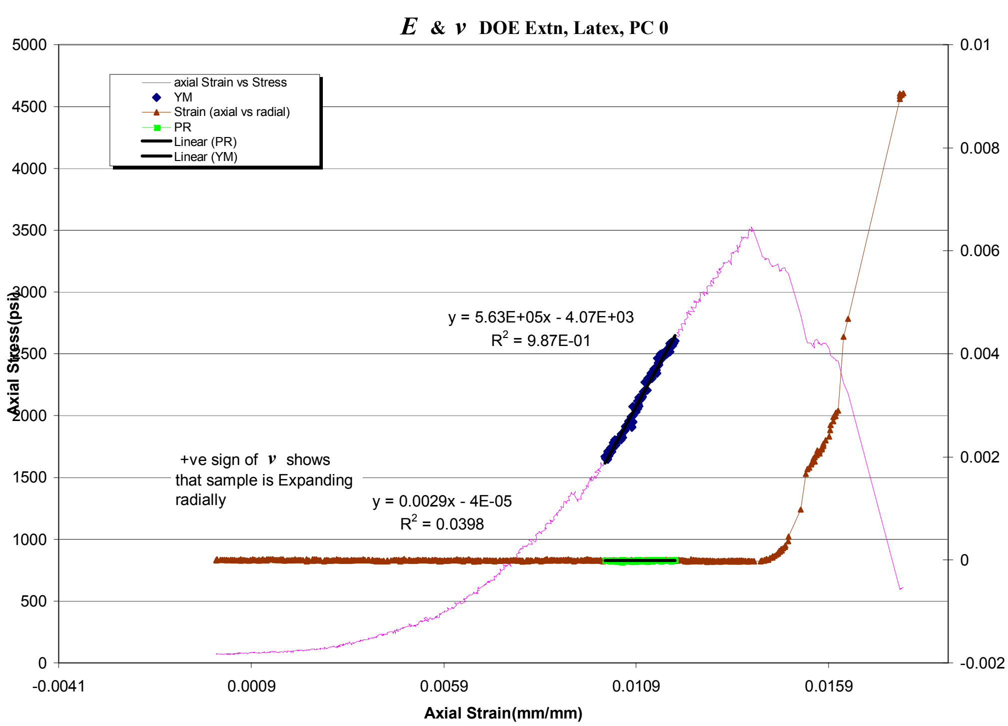


**Figure B14— Plot of compressive Young's modulus for latex slurry at 250-psi confining pressure.**

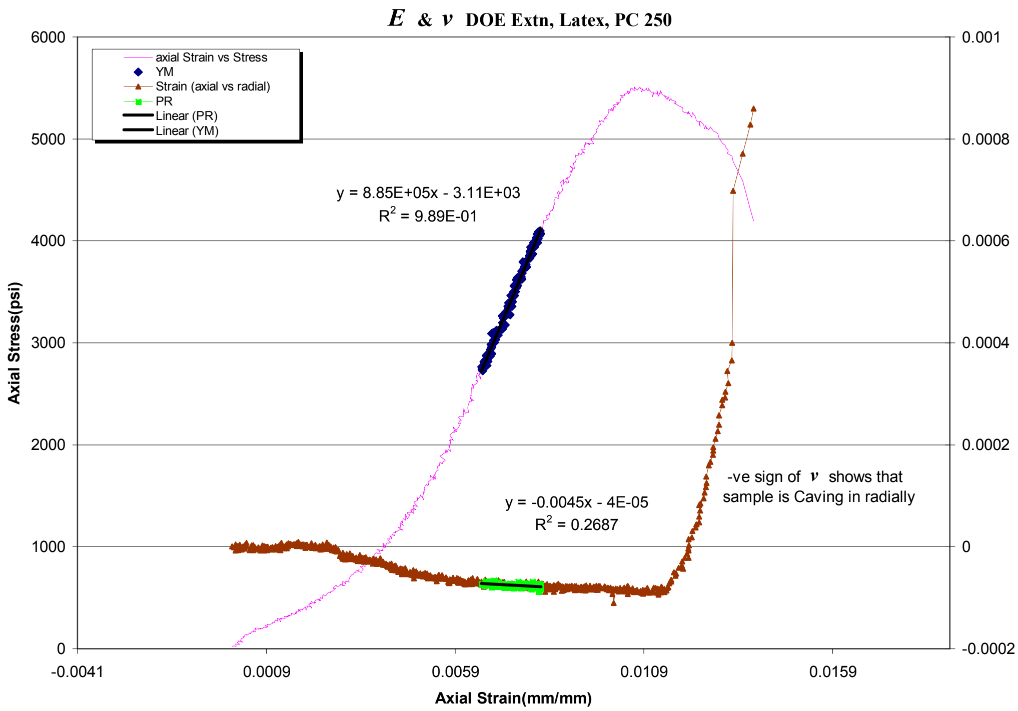



**Figure B15— Plot of compressive Young's modulus for latex slurry at 500-psi confining pressure.**

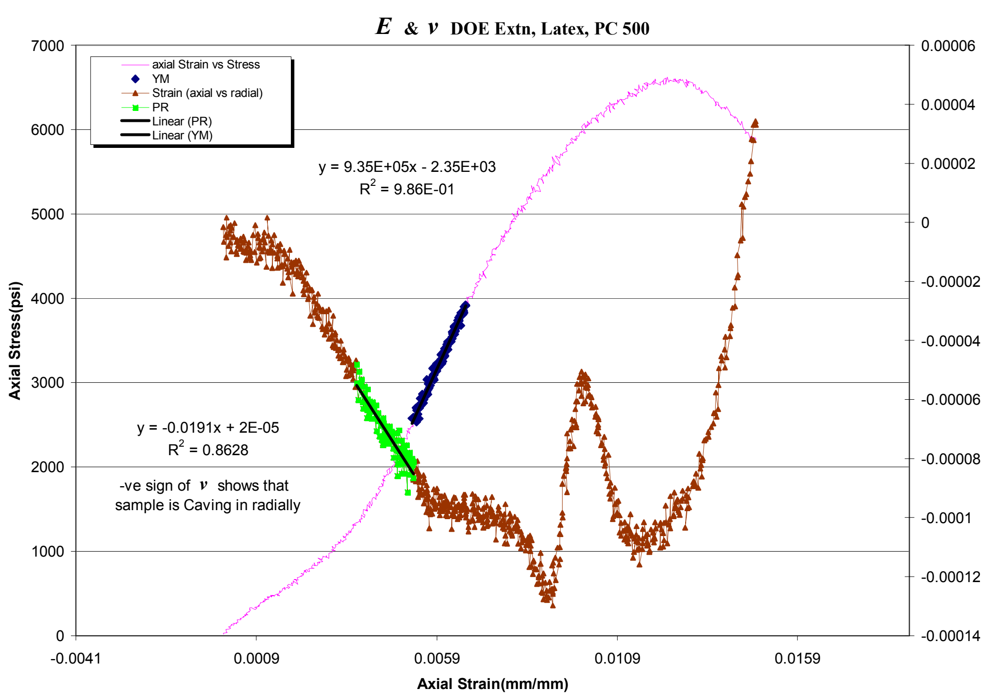


**Figure B16—Young's modulus measurements for Type 1 slurry at 500-psi confining stress and a 100-psi/min load rate.**

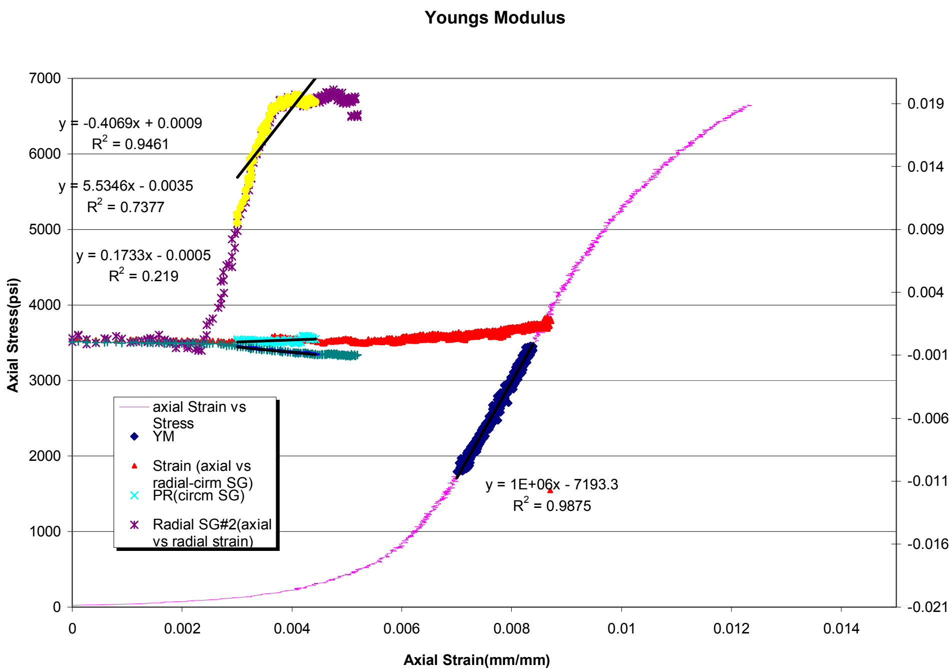



**Figure B17—Young's modulus measurements for Type 1 slurry at 500-psi confining stress and a 250-psi/min load rate.**

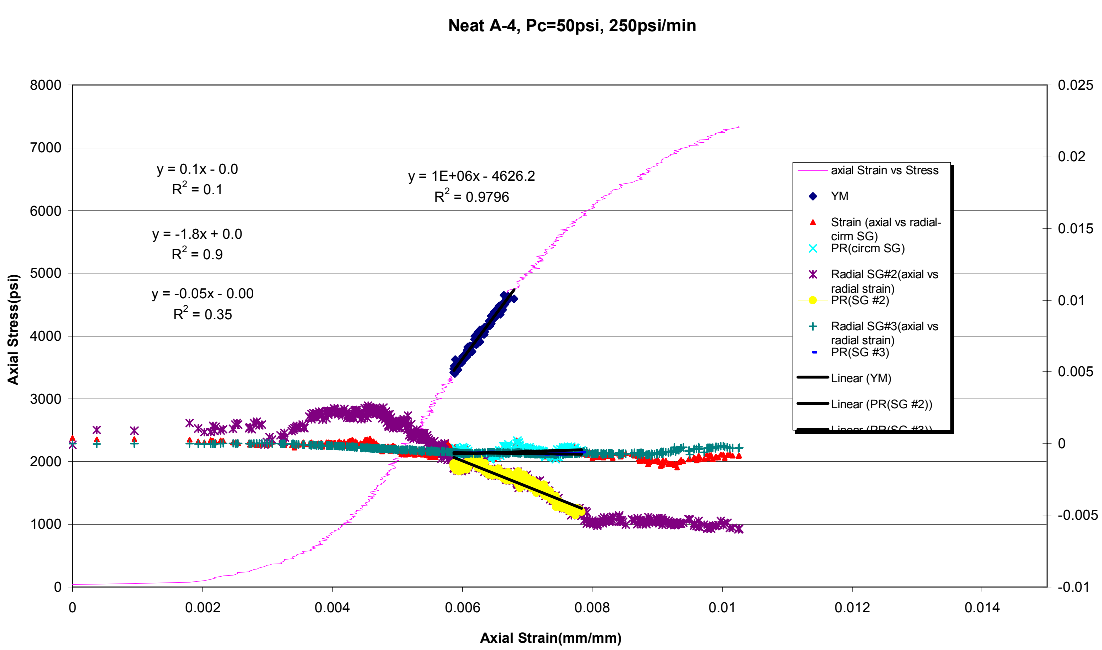


**Figure B18—Young's modulus measurements for Type 1 slurry at 500-psi confining stress and a 500-psi/min load rate.**

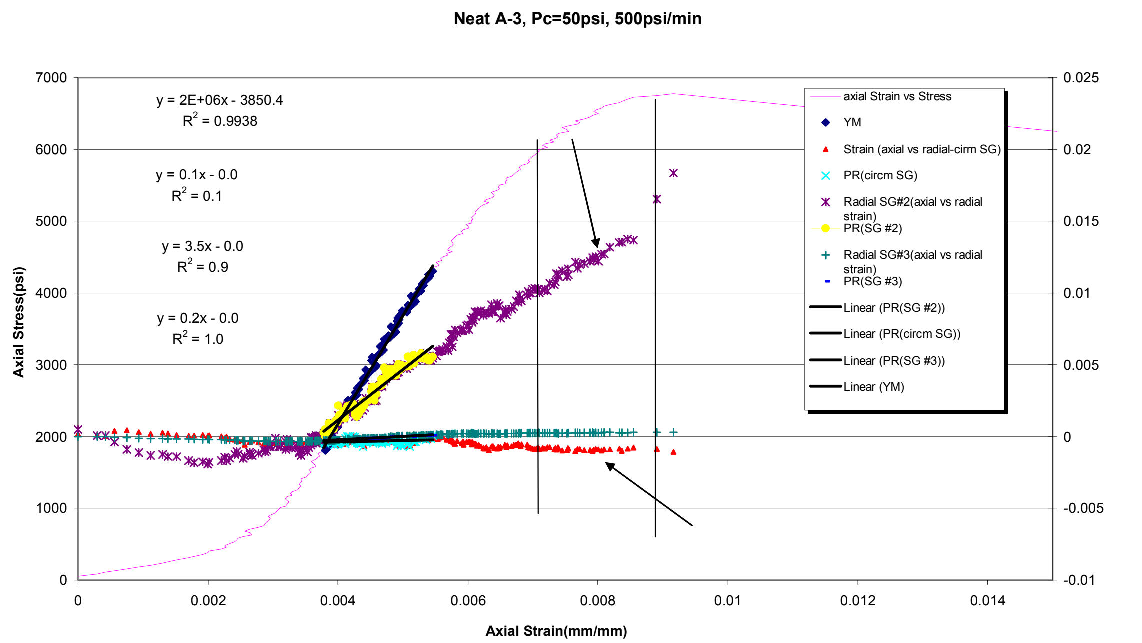
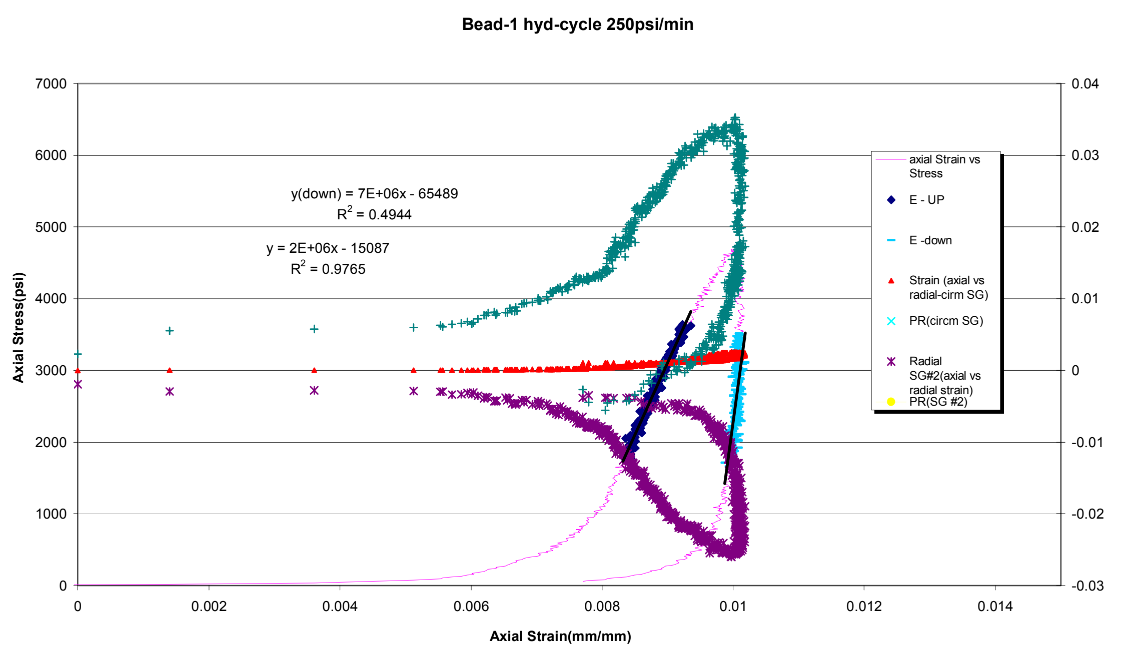
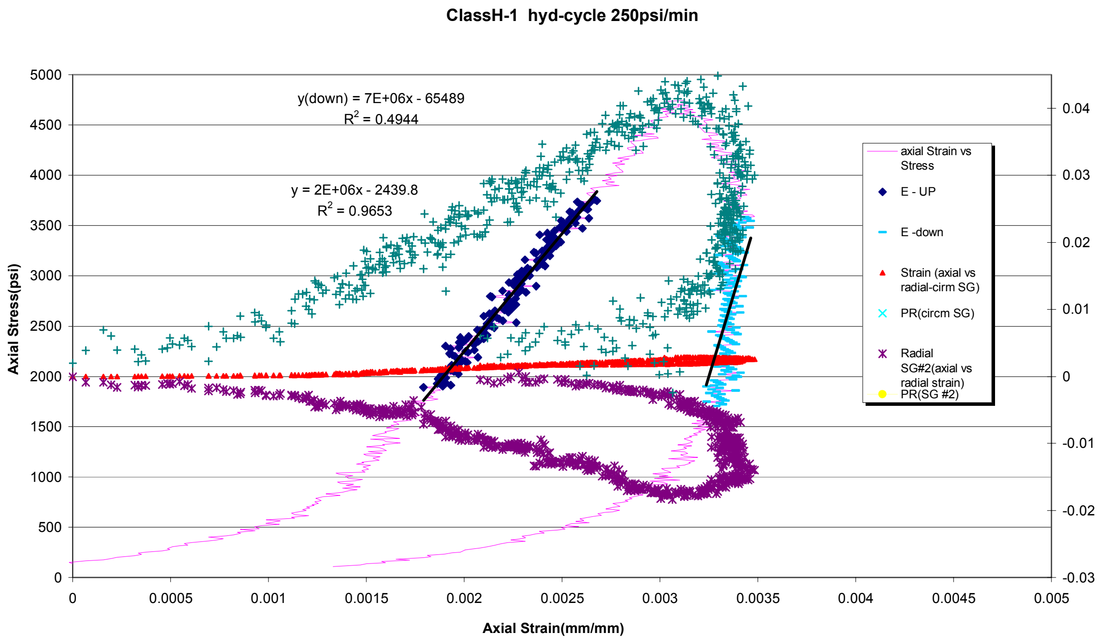



**Figure B19—Hydrostatic cycling data for bead slurry showing anelastic strain.**



## Figure B20— Hydrostatic cycling data for Class H slurry showing anelastic strain.




## Figure B21— Hydrostatic cycling data for 12-lb/gal foam slurry showing anelastic strain.

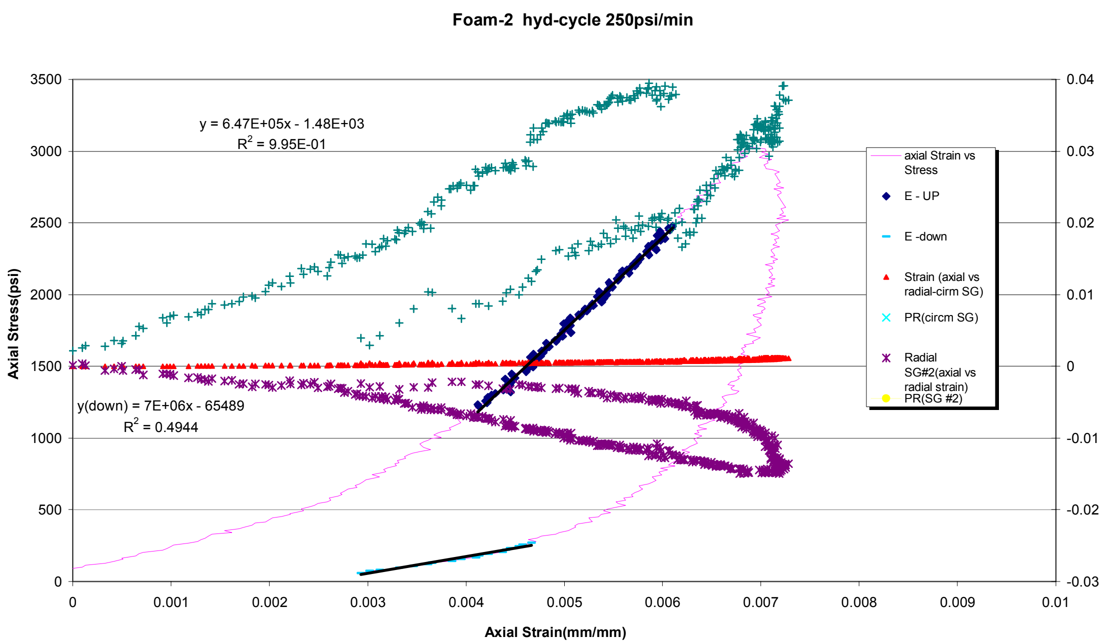


**Figure B22— Hydrostatic cycling data for Type 1 slurry showing anelastic strain.**

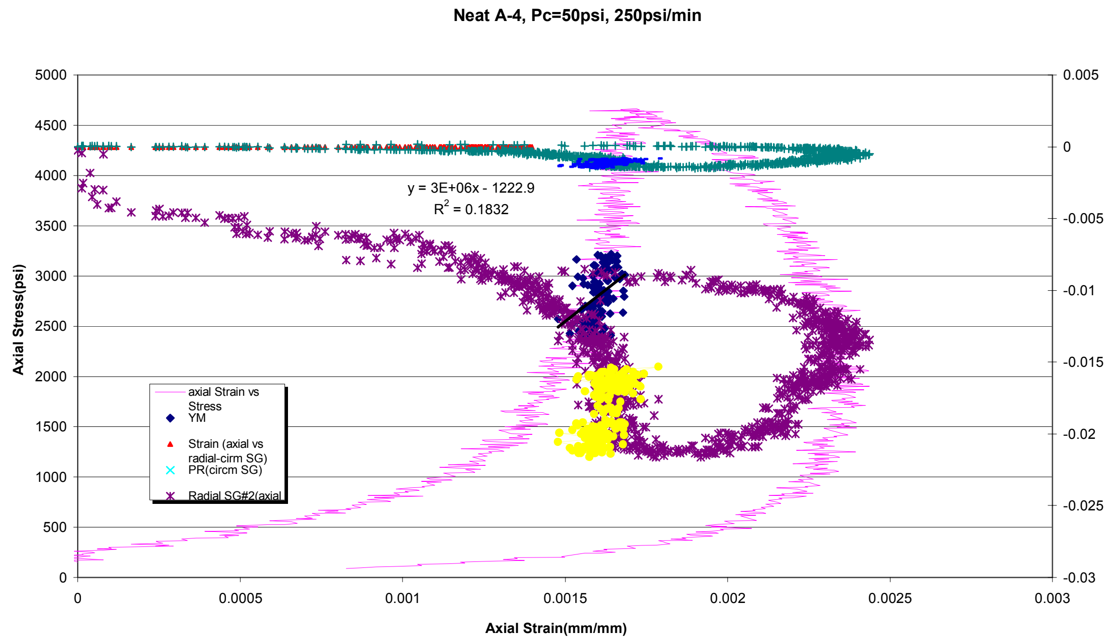



**Figure B23— Hydrostatic cycling data for sodium metasilicate (SMS) slurry showing anelastic strain.**

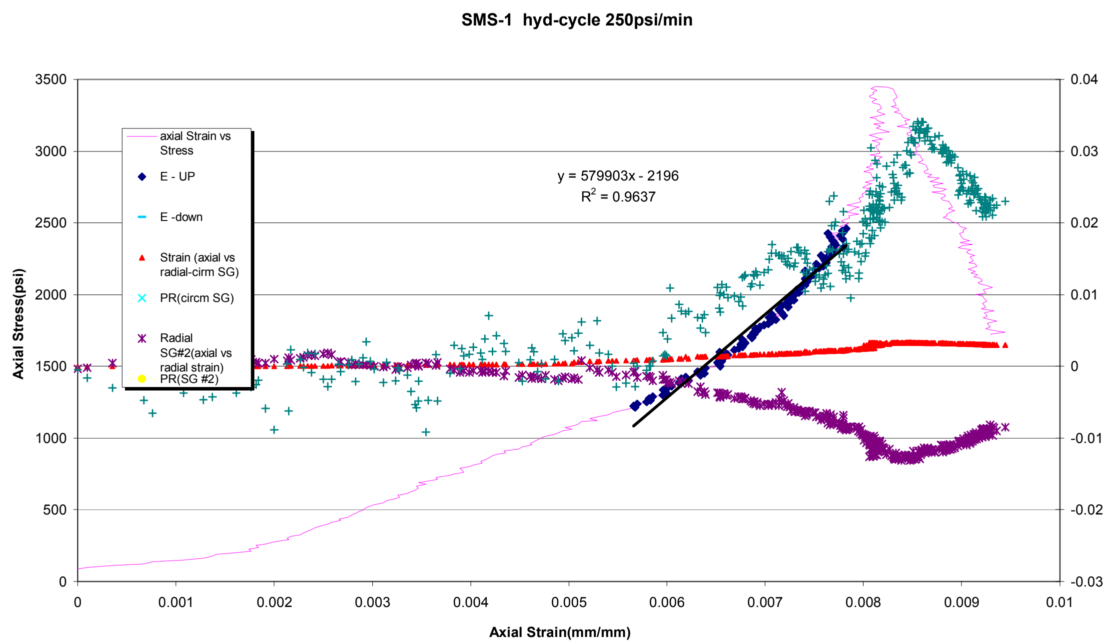


**Figure B24— Anelastic strain failure load for neat Type 1 slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

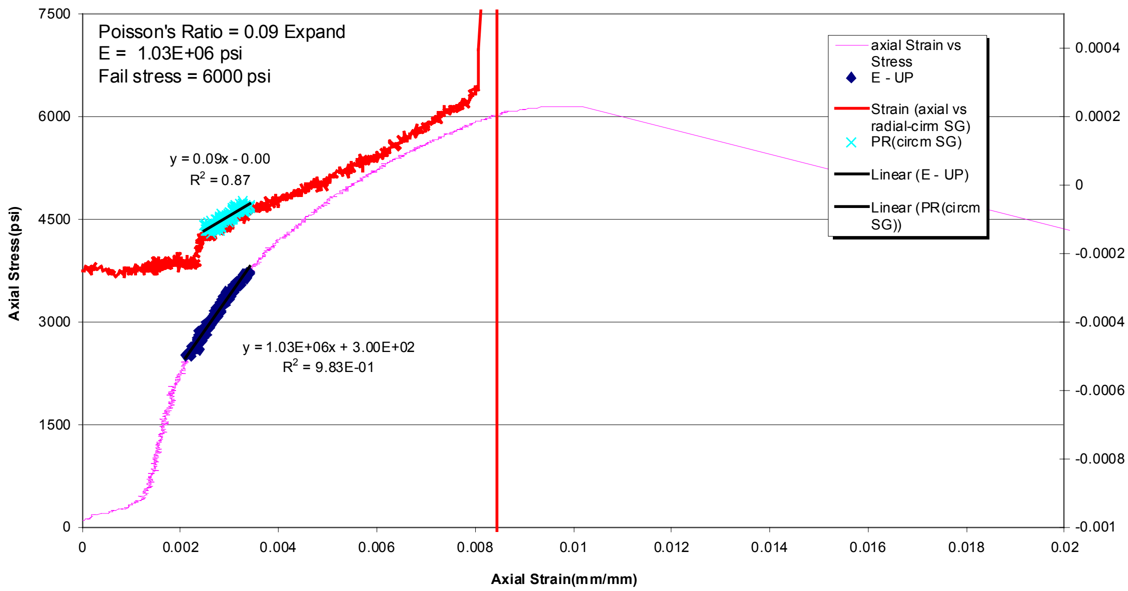



**Figure B25— Anelastic strain failure load for foam slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

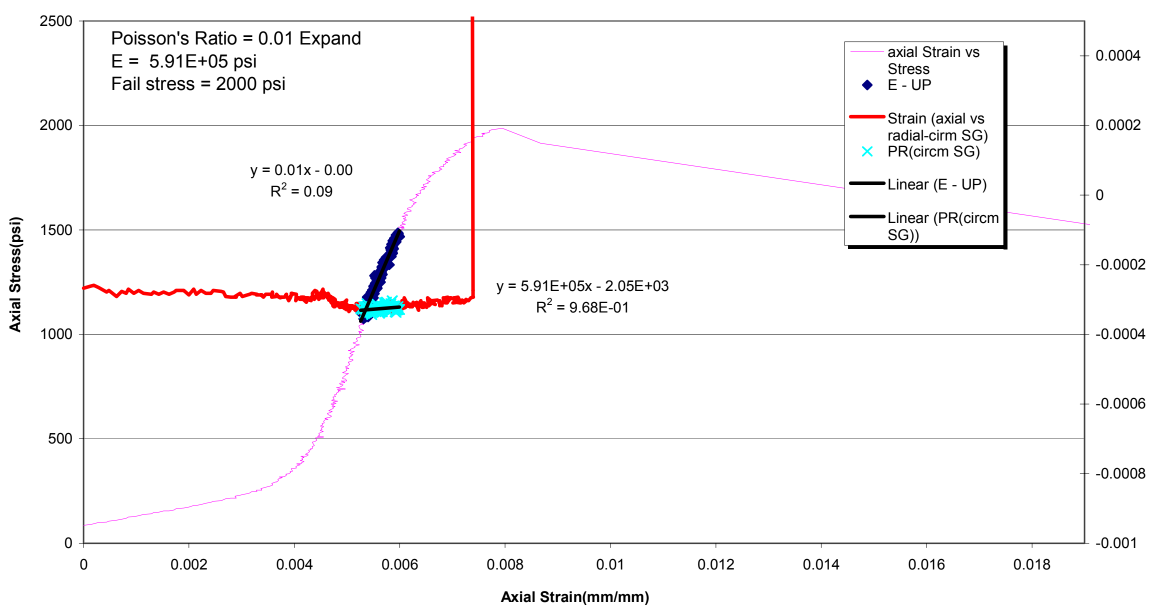


**Figure B26— Anelastic strain failure load for bead slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

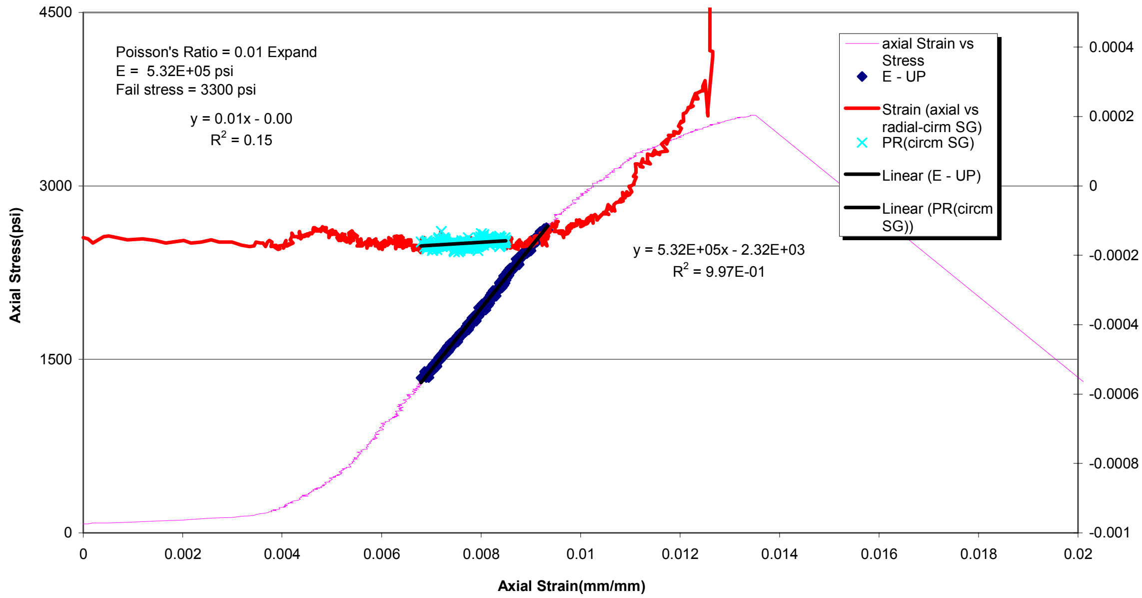



**Figure B27—Anelastic strain failure load for latex slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

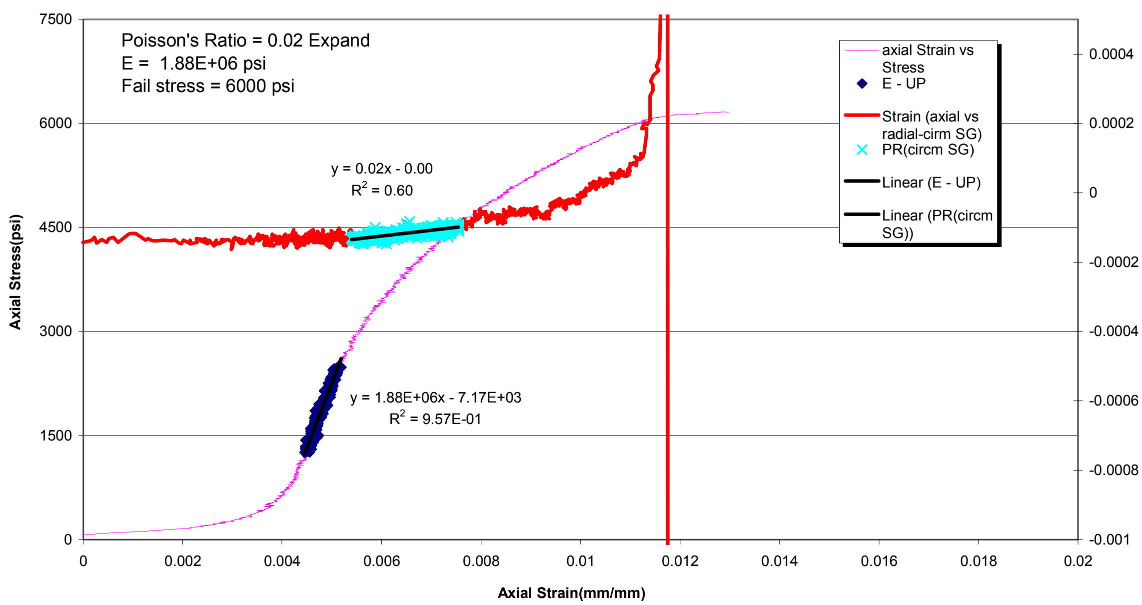


**Figure B28—Anelastic strain, cycled to 25% of failure load, for Type 1 slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

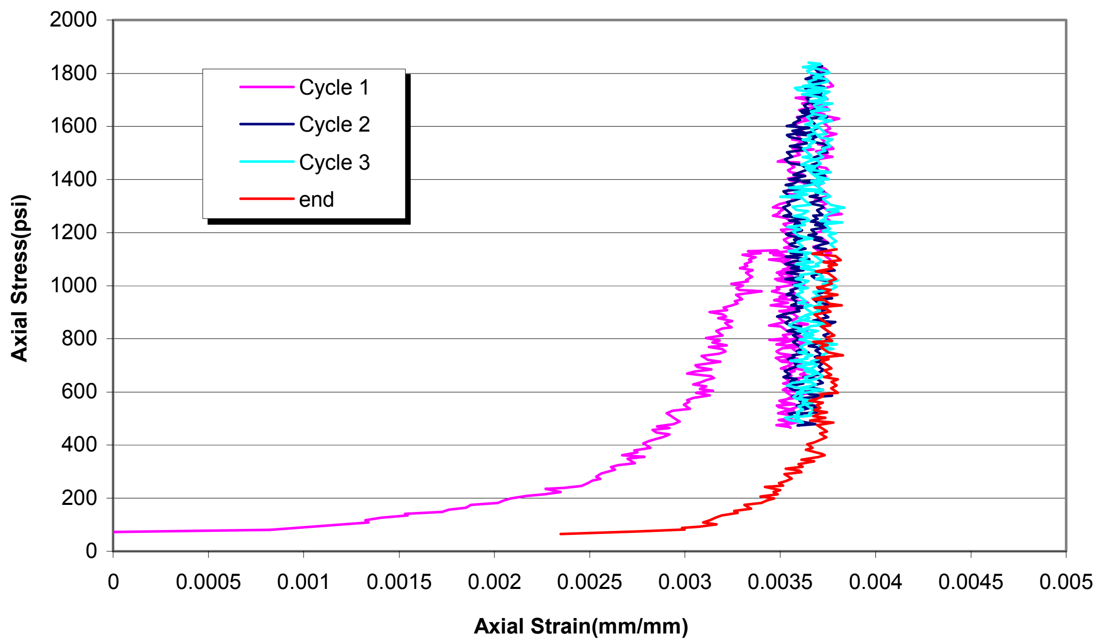



**Figure B29—Anelastic strain, cycled to 25% of failure load, for foam slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

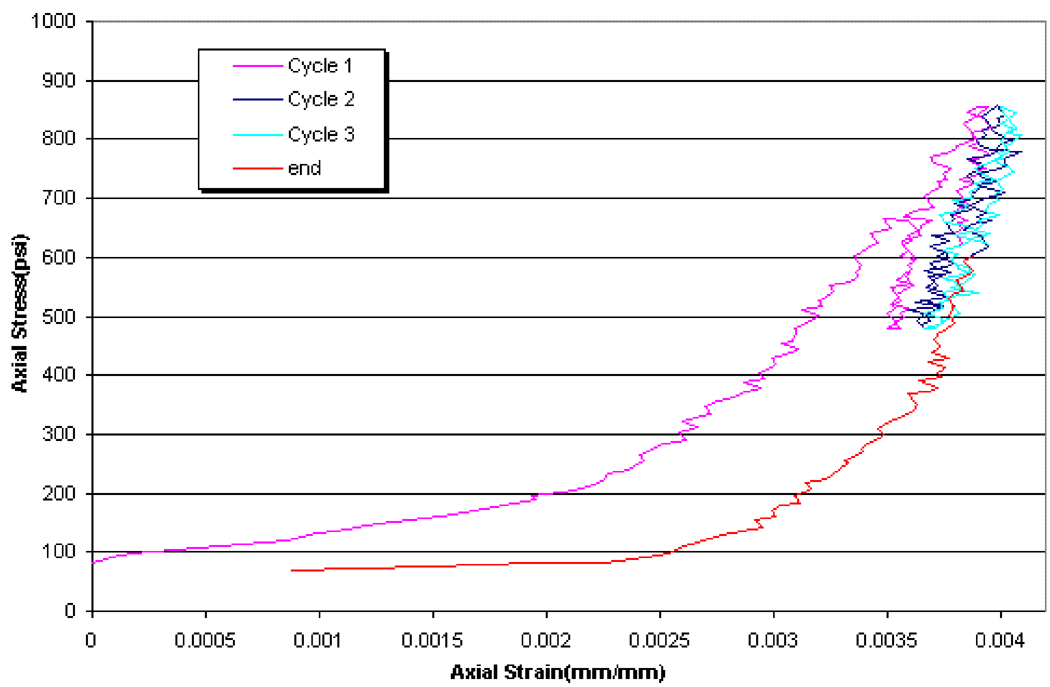


**Figure B30—Anelastic strain, cycled to 25% of failure load, for bead slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

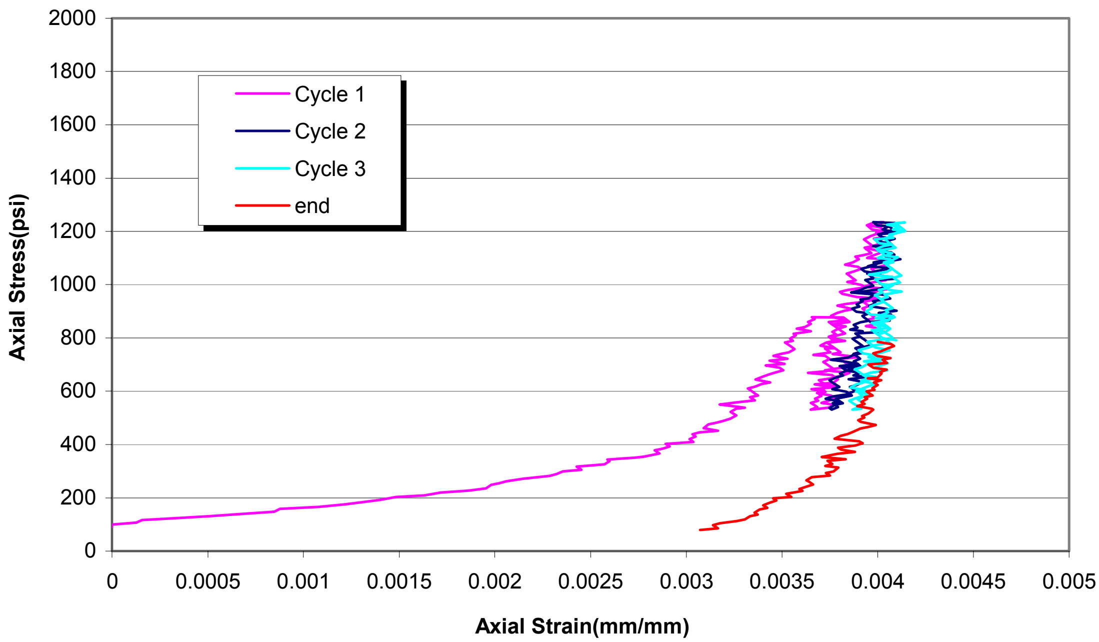



**Figure B31—Anelastic strain, cycled to 25% of failure load, for latex slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

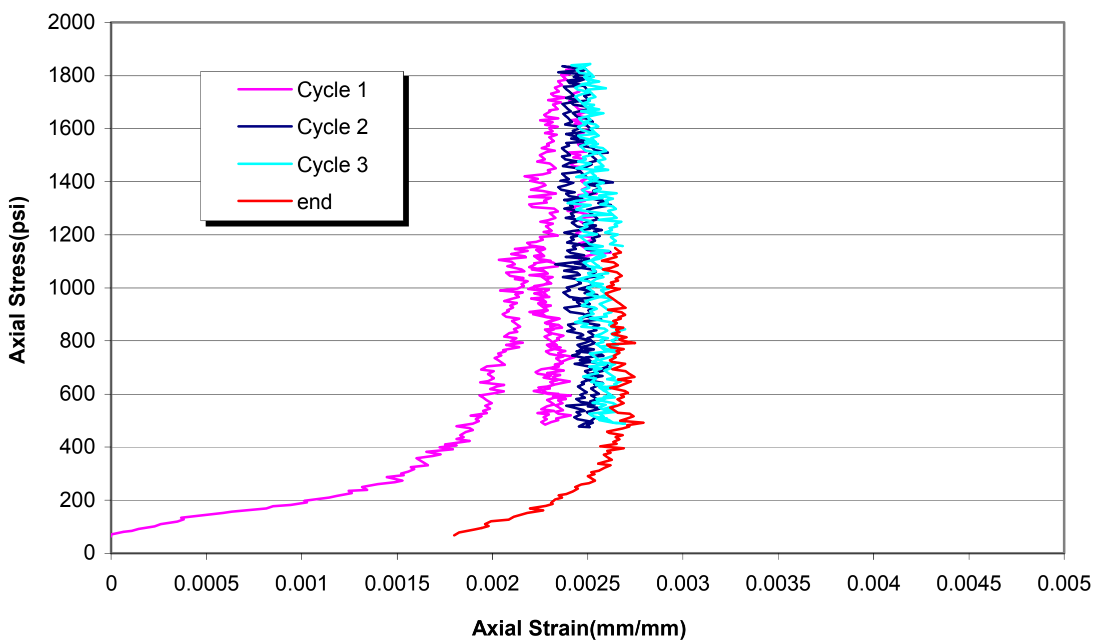


**Figure B32—Anelastic strain, cycled to 50% of failure load, for Type 1 slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

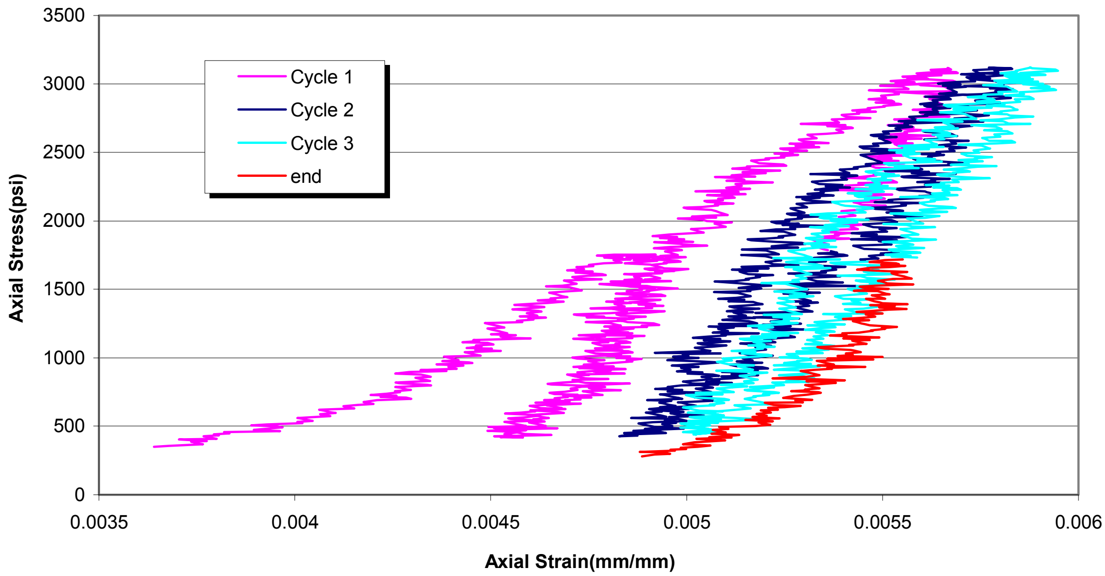



**Figure B33—Anelastic strain, cycled to 50% of failure load, for latex slurry at a load rate of 250 psi/min and confining pressure of 500 psi.**

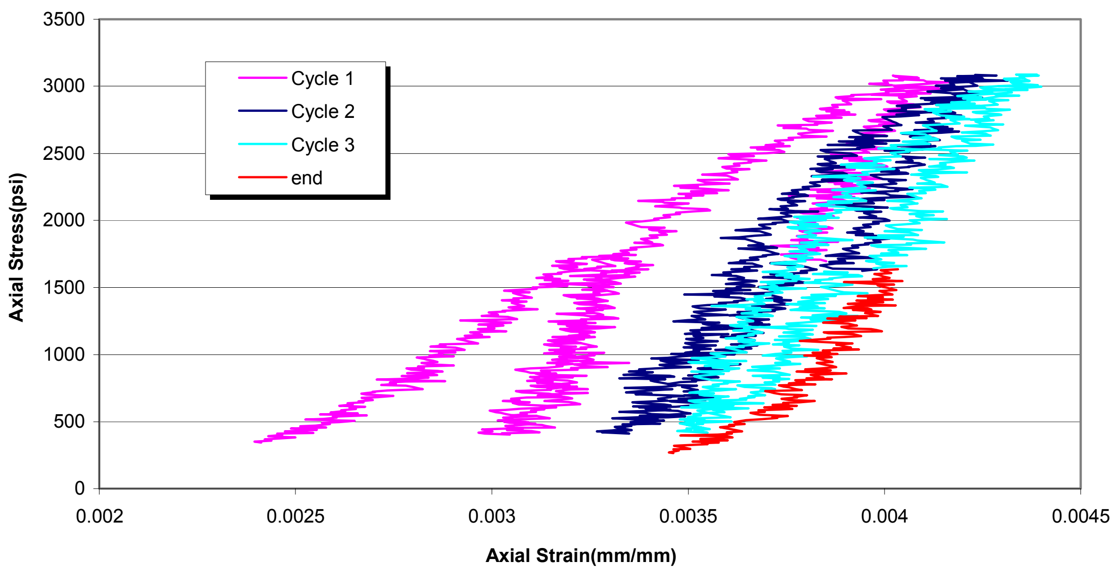


**Table B1—Chronicle of 8-ft Permeability Model Testing (cc/min)**

| | Days Tested | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **Slurry** | **1** | **7** | **14** | **23** | **37** | **44** | **51** | **60** | **63** | **65** | **66** |
| Type 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | 0 | 0 | 0 | 0 | 0 | 0 | 0.107 | 0.12 | 0.116 | 0.05 | 0.05 |
| SMS | 33 | 71 | 72 | 70 | 71 | 71 | * | * | * | * | * |
| | 26 | 57 | 60 | 42 | 30 | 30 | * | * | * | * | * |
| Bead | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Latex | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | Days Tested | | | | | | | | | | |
| **Slurry #** | **67** | **71** | **73** | **78** | **79** | **80** | **84** | **85** | **86** | **87** | **88** |
| Type 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | 0.05 | 0 | 0.05 | 0.03 | 0.03 | 0.02 | 0 | 0 | 0 | 0 | 0 |
| SMS | * | * | * | * | * | * | * | * | * | * | * |
| | * | * | * | * | * | * | * | * | * | * | * |
| Bead | 0 | 0 | 0 | 0.03 | 0.03 | 0.03 | 0.02 | 0.02 | 0.02 | 0.02 | 0.02 |
| | 0 | 0 | 0 | 0 | 0 | 0 | 0.02 | 0.02 | 0.02 | 0.02 | 0.02 |
| Latex | 0 | 0 | 0 | 0 | 0 | 0 | 0.02 | 0.02 | 0.02 | 0.02 | 0.02 |
| | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | Days Tested | | | | | | | | | | |
| **Slurry #** | **99** | **100** | **101** | **105** | **106** | **107** | **108** | **113** | | | |
| Type 1 | 0 | 0 | 0 | 0 | 0 | 0.09 | 0.08 | 0.11 | | | |
| | 0 | 0 | 0 | 0.23 | 0.217 | 1.3 | 1.24 | 1.71 | | | |
| SMS | * | * | * | * | * | * | * | * | | | |
| | * | * | * | * | * | * | * | * | | | |
| Bead | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | |
| | 0.02 | 0.02 | 0.02 | 0 | 0 | 0.01 | 0 | 0 | | | |
| Latex | 0.6 | 0.8 | 0.8 | 0.74 | 0.87 | 2.75 | * | * | | | |
| | 3.1 | 3.51 | 3.51 | 3.51 | * | * | * | * | | | |

| | | | | |
|---|---|---|---|---|
| Day 1 Thru 44 - 100 PSI | Day 51 - 200 PSI | Day 60 Thru 73 - 300 PSI | Day 78 Thru 88 - 400 PSI | |
| Day 88 Thru 113 - 500 PSI | | | | |

[1] API Recommended Practice 10B: "Recommended Practice for Testing Well Cements," 22nd Edition, American Petroleum Institute, Washington, D.C., December 1997.

[2] ASTM C469, Standard Test Method for Static Modulus of Elasticity (Young's Modulus) and Poisson's Ratio of Concrete in Compression.

[3] "Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens," ASTM C496-96, West Conshohocken, PA, 1996.